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General and Physical Chemistry.

Influence of Pressure on the Index of Refraction of Water for Sodium Light. By L. ZEHNDER (*Ann. Phys. Chem.* [2], **34**, 91—121).—This investigation was carried out principally with the view of deciding whether the so-called specific refraction or some similar expression was truly a constant. Gladstone and Dale (*Phil. Trans.*, **153**, 317) have investigated the effect of change of pressure on the index of refraction of liquids, and they came to the conclusion that when D is the density of the liquid, the quantity $(\mu - 1)/D$ is a constant. Landolt (*Ann. Phys. Chem.*, **123**, 595) arrived at a similar conclusion. Rühlmann (*ibid.*, **132**, 1) and Wüllner (*ibid.*, **133**, 1) came to the conclusion that it was not perfectly constant. According to Jamin (*Ann. Chim. Phys.*, **52**, 163), the quantity $(\mu^2 - 1)/D$ is a constant. Mascart (*Ann. Phys. Chem.*, **153**, 154) denied the constancy of this quantity, and Quincke (*ibid.* [2], **19**, 401) confirmed Gladstone and Dale's conclusion. L. Lorentz (*ibid.* [2], **11**, 70) and H. A. Lorentz (*ibid.* [2], **9**, 641) independently arrived at the result that $(\mu^2 - 1)/(\mu^2 + 2) \cdot 1/D$ was constant.

The author, as the results of a series of observations carried out with minute precautions, arrives at the conclusion that Gladstone and Dale's result, $(\mu - 1)/D$ = constant, is a very close approximation to the truth, and that the other relations are not.

G. W. T.

Spectra of Oxygen. By J. JANSSEN (*Compt. rend.*, **106**, 1118—1119).—Observations on the Pic du Midi show that the indistinct absorption-bands in the oxygen spectrum (Abstr., 1886, 749) can be seen in the solar spectrum if the atmospheric layer through which the sunlight passes is of sufficient diameter. The existence of these bands, and the law that their intensity is proportional to the product of the diameter of the layer of oxygen into the square of the density of the gas, is confirmed by a series of observations extending from normal pressure to a pressure of 100 atmos. with tubes varying from 0·42 to 60 metres in length. The author calculates that these bands should be just visible with a layer of liquid oxygen 4 to 5 mm. in thickness; and Olzewski has actually observed them with a layer of 7 mm. in thickness.

C. H. B.

Spectrum of Gold. By E. DEMARÇAY (*Compt. rend.*, **106**, 1228—1230).—The lines at 5230, 4437, 4338, and 4064, which Boisbaudran observed in the spectrum of gold, but which Krüss attributes to the presence of platinum and palladium, are not really due to these metals. They are visible although all the other lines of platinum and palladium are absent, and they do not actually coincide with

any lines of these metals. The lines 5230, 4437, and 4064 are undoubtedly due to gold, but the author was unable to observe the line 4338 in a condensed spark spectrum.

Boisbaudran has since found that the line 4338 is only visible with concentrated solutions of gold chloride, and it may be due to a non-metallic element. C. H. B.

Application of the Method of High Interferences to Quantitative Spectrum Analysis. By H. EBERT (*Ann. Phys. Chem.* [2], **34**, 39—90).—The author points out that the researches of Fizeau and Foucault (*Ann. Chim. Phys.* [3], **26**, 138, and [3], **66**, 429; *Compt. rend.*, **21**, 1155, **26**, 680, and **54**, 1237), Billet (*Compt. rend.*, **67**, 1000), Mascart (*Ann. de l'école Normal* [2], **1**, 57), Ketteler (*Beobachtungen über die Farbenzerstreuung der Gase*, Bonn, 1865), Müller (*Ann. Phys. Chem.*, **150**, 86), the author himself (*ibid.* [2], **32**, 337), and Michelson and Morley (*Amer. J. Sci.* [3], **34**, 427), on interference with large difference in length of path, show that although the possibility of interference is limited by conditions depending on the change in the vibrations of the luminous particles, the limits are wide ones, and the last-named authors observed cases of interference with a difference in length of path of as much as 200,000 times the wave-length. Müller was the first who applied this to the study of the source of light itself.

The method is very suitable for investigating small alterations in the refrangibility of lines in the spectrum. With light corresponding with the green mercury line, the author has obtained interference phenomena with a difference in length of path 80,000 times the wave-length, and as a shifting in one of the bands of one-tenth its thickness can be observed, an alteration of 1/800000 of the wave-length, or 1/800 of the distance between the two sodium lines can be determined.

The most homogeneous lines in the spectrum are formed by a number of component vibrations which differ very little in wave-length, and the amplitudes of which are a definite function of the wave-length; alterations in the interference bands simply giving evidence of changes in the mean wave-length.

The determination of the greatest difference in length of path for which interference bands are obtained gives a measure of the time during which the luminous molecules maintain their vibrations unchanged, because two rays from the same luminous particle will interfere only if the vibration is the same at the moment of emission of each of the rays.

Wiedemann has considered this question (*Ann. Phys. Chem.* [2], **5**, 503; *Phil. Mag.* [5], **7**, 77), and has shown that the value of this maximum length of path is intimately connected with the mean length of the molecular free path, and in the case of sodium vapour the method gave a number of the same order of magnitude as that calculated from the kinetic theory of gases.

An increase in the density of the gas will narrow the limits within which interference is possible, as after contact between two molecules the vibrations will be altered, so that two rays to interfere must both

come from a molecule during the interval between two encounters. Lippich has also shown (*Wien. Ber.*, **82**) that an increase of temperature has the same effect, but the increase of the density is of greater importance, as is easily seen, and was pointed out by Wiedemann in the paper referred to. This physicist has applied the method to the investigation of the pressure at the surface of the sun and stars (*Phil. Mag.* [5], **10**, 123).

It is clear from these considerations that the method can only give a lower limit for the duration of the undisturbed vibration of a free molecule. An important question to be determined is the influence exerted by the breadth of the spectrum line on the greatest length of path for which interference is possible.

Müller, in the paper referred to, shows that the finite breadth of a spectrum line gives rise to secondary systems of interference bands, causing an alteration in the clearness of the primary bands, and he shows by theoretical reasoning that the effect of gradually widening the line, maintaining the difference in length of path constant, has the same effect as gradually increasing the difference in length of path while maintaining the breadth constant. In confirmation of this he states that when a bead of salt is gradually plunged deeper into a bunsen flame, thus increasing the quantity of vapour, the lines become wider. The author has been unable to observe this alteration, and believes that Müller must have been mistaken, as the effect could not possibly be sufficient to be appreciated by the eye. The author does find an increase in clearness when the bead is plunged deeper into the flame, but only when it passes into the cooler interior portion, so that the quantity of vapour is diminished, and therefore the spectrum line becomes more homogeneous.

With regard to the limits of the method, the author shows that it is impossible to follow the shifting of the bands due to broadening of the line for much more than the width of the band, but the extent to which the widening of a line for a given difference in length of path can be observed is only limited by the thinness of the plates used to obtain the fringes; and on the other hand, by increasing the thickness of the plates, the earlier stages of the widening process can be observed.

The authors made experiments with beads consisting of salts of lithium, potassium, strontium, and sodium. When the bead first touched the outside of the flame the illumination was feeble, and the interference bands became sharply defined. As the bead was plunged more deeply into the flame the bands became less distinct, and the illumination of the field of view increased, the lines being widened and the light becoming less homogeneous. The depth to which the bead had to be plunged into the flame to cause complete disappearance of the bands was greater for the less fusible salts, and for those which gave the least homogeneous light. When the bead reached the cool interior portion of the flame, the bands reappeared. This alternate appearance and disappearance of the bands are in the author's opinion the explanation of the alternation described by Müller.

As the illumination gradually increased, the bands appeared to remain constant in position until at a certain point, corresponding with

the maximum formation of vapour, they were suddenly shifted. This sudden widening of the spectrum line may be compared with some somewhat similar phenomena observed by Cornu (*Ann. Chim. Phys.* [6], **7**, 5).

The displacement of the bands was in the same direction, but not of the same amount for all the lines of the spectrum which were observed. An increase in the quantity of vapour caused a displacement indicating a decrease in the refrangibility of the widened line, so the widening was greater towards the least refrangible end. The displacement was not perceptibly altered by altering the thickness of the plate, showing that it is independent of the difference in the length of path, and depends only on the variation in brightness of the different constituent portions of the band.

With regard to the causes of the widening of the line, as the bead was gradually introduced into the flame, a change must take place (1) in the thickness of the luminous layer, (2) in the temperature, (3) in the density of the luminous vapour, (4) in the nature and energy of chemical decomposition. As a result of these, there would also be a variation in the intensity of illumination, but the author's previous researches (*Ann. Phys. Chem.* [2], **32**, 337) have shown that this by itself does not alter the wave-length. The thickness of the layer was limited by the thickness of the mantle of the flame, which was about 8 mm., and within these limits its variation was found to be without influence on the mean refrangibility of the spectrum line. Alteration in the temperature did not cause any displacement in the bands, although considerable ranges of temperature were obtained, and the author concludes from his experiments that spectrum lines when beginning to widen through increase of temperature, until a certain breadth has been passed, do not undergo a change in mean refrangibility of more than a millionth part within the limits of the experiments, in which the temperature was increased by about one-third of its lowest value. The author arrives at the conclusion that the observed asymmetric widening of the lines is due entirely to the increase in the density of the vapour.

With regard to the influence of the chemical decompositions, he finds that if it exists at all it must be exceedingly small.

G. W. T.

Relation between the Constitution and Specific Rotatory Power of Organic Compounds. By B. Sorokin (*J. pr. Chem.* [2], **37**, 320—333).—The author finds that the same solvent produces identical changes in the specific rotatory power of closely related organic compounds. When, for example, the anilide of dextrose is dissolved in a 90 per cent. solution of ethyl alcohol, $[\alpha]_D = -44{\cdot}11°$, whilst for a solution in methyl alcohol $[\alpha]_D = -48{\cdot}73°$. The toluide of dextrose in a 90 per cent. solution of ethyl alcohol gives $[\alpha]_D = -38{\cdot}80°$, whilst a solution in methyl alcohol shows $[\alpha]_D = -43{\cdot}88°$. The combination of an aniline or toluidine molecule with a molecule of dextrose produces identical changes in its optical activity. Tables showing the specific rotatory power of salicin, helicin, and other related substances are given.

G. T. M.

Conduction of Electricity through Gases. By R. Nahrwold (*Ann. Phys. Chem.* [2], **34**, 170—172).—Narr (this vol., p. 397) ascribes to the author the statement that the molecules of a gas are incapable of receiving a static charge. This the author says is incorrect. He concluded from his experiments that it was impossible to give an electrostatic charge to a mass of gas, but drew no conclusion as to the possibility of separate molecules becoming charged, which is quite possible, as the charge of the molecules might very possibly be dissipated before an observation could be made, either by dissociation or otherwise.

He also states that although in an earlier paper than the one quoted by Narr he alluded to the possibility of the charge on a body surrounded by a gas being dissipated by the action of dust particles, he inclines to the belief that conduction does actually take place through gases, even when no dust particles are present; and he quite agrees with Narr that his investigations leave it an open question.

G. W. T.

New Apparatus for Electrochemical Research. By N. v. Klobukoff (*J. pr. Chem.* [2], **37**, 375—381).—The author describes and figures a "stopper" rheostat and an electrode holder, the chief feature in both being that contact is made by means of mercury.

A. G. B.

Alternate Currents and Electrolytes. By S. Sheldon (*Ann. Phys. Chem.* [2], **34**, 122—138).—Two essentially distinct methods are in use for determining the electrical resistance of liquids, namely, by means of constant currents, and by means of alternating currents. In order to compare the two methods, the author made a series of measurements by both methods of the resistances of dilute solutions of potassium chloride, sodium chloride, potassium sulphate, and magnesium sulphate, of nine different degrees of concentration. In every case, the results obtained by the two methods were closely concordant, and the author concludes that both the methods, if properly carried out, are capable of giving equally good results, but he decides in favour of the alternate current method, on the grounds of simplicity and convenience.

G. W. T.

Electrolytic Formation of Persulphuric Acid and Hydrogen Dioxide at the Anode. By F. Richarz (*Ber.*, **21**, 1669—1674, and 1682—1683; compare this vol., p. 12).—A reply to Traube (this vol., p. 210).

F. S. K.

Constitution of Peroxides. By F. Richarz (*Ber.*, **21**, 1675—1681).—A reply to Traube's theory of the "oxygen molecule-compounds" (this vol., p. 210), in which the author endeavours to show that Helmholtz's electrochemical theory explains the behaviour of ozonides and antozonides (peroxides) without the aid of any new hypothesis.

F. S. K.

Magnetism of Organic Compounds. By S. Henrichsen (*Ann. Phys. Chem.* [2], **34**, 180—221).—The substances selected by the author for his experiments were all of the fatty series, these com-

pounds being chosen because of the large number of homologous series and isomeric compounds which they contain, and also because a very large number of them can be obtained in a state of purity with very little trouble and expense. The researches were all made with liquids, as being easier to work with than either gases or solids. The author found that—1. All the substances experimented on were diamagnetic. 2. For every CH_2-group introduced into the formula, the molecular magnetism increases by a quantity the mean value of which is 16·32 times that of the molecular magnetism of water. 3. The values of the molecular magnetism are equal for isomeric and metameric substances, provided the nature of the union is the same in each substance. 4. The molecular magnetism depends on the manner in which the atoms are united. A double bond appears to diminish the molecular magnetism. 5. The specific magnetism of primary and normal compounds is greater than that of secondary and iso-compounds, similarly it is greater for acids than for the corresponding ethereal salts. 6. By making certain assumptions the values of the atomic magnetism of the individual elements may be calculated from the experimental data. The values obtained are—

H.	O′.	O″.	C′.	C″.	Cl_r.	Cl_{rr}.	Cl_{rrr}.
9	129	−17	145·2	98	282	249	218

Cl_{rrrr}.	Br_r.	Br_{rr}.	Br_{rrr}.	I.	I_r.	S_r.
194	413	374	334	642	577	284

7. The values of the atomic magnetism of the halogens decrease as the number of atoms increases (the number being indicated by the dashes below the symbols in the table).

The nature of the assumption under which the results in (6) were obtained may be illustrated by giving the method of obtaining them for oxygen in single or double union respectively:—Consider compounds containing only carbon, oxygen, and hydrogen, namely, the alcohols, aldehydes, acids, and ethereal salts. The observations give the value of the molecular magnetism for each of these groups, and also the value for CH_2; four equations are therefore obtained which will serve to determine four quantities. Let it now be assumed that in these compounds all the carbon- and hydrogen-atoms have the same molecular magnetism, but that the value for oxygen united by one bond to carbon, O′, differs from that of O″, oxygen united by two bonds. The alcohols can then be represented by the formula $n(CH_2)HO'$, the aldehydes by $n(CH_2)O''$, and the acids and compounds of alcohol radicles by $n(CH_2)O'O''$. The value for O′ can then be determined directly as follows:—

Aldehyde,
$C_2H_4O'' - C_2H_4 = O''$.
307 − 326 = −19

Acetone,
$C_3H_6O'' - C_3H_6 = O''$.
473 − 490 = −17

Isobutaldehyde,
$C_4H_8O'' - C_4H_8 = O''$.
640 − 653 = −13

Valeraldehyde,
$C_5H_{10}O'' - C_5H_{10} = O''$.
797 − 816 = −21

From the previous determination of the value of C_2H_5, the most probable value is found to be $O''=-17$, the negative sign indicating that doubly-bound oxygen is paramagnetic. Again—

Formic acid,	Acetic acid,
$CH_2O'O''-CH_2 = O'O''$.	$C_2H_4O'O''-C_2H_4 = O'O''$.
$275 - 163 = 112$	$437 - 326 = 111$

Propionic acid,
$C_3H_6O'O''-C_3H_6 = O'O''$.
$591 - 490 = 101$

The most probable value is—

	$O'O''$	$= 112$
And we had..	O''	$= -17$
Therefore....	O'	$= 129$

G. W. T.

Specific Heats of Liquid Carbon Compounds. By R. Schiff (*Zeit. physikal. Chem.*, **1**, 376—390; compare Abstr., 1887, 6).—The author has extended his investigations to fresh series of compounds, and obtained results similar to those previously arrived at. In each case, the mean specific heat between the temperatures t and t' may be expressed by $C_{t-t'} = a + b(t + t')$, or the true specific heat by $K_t = a + 2bt$, the coefficient b having a practically constant value for any homologous series, and a having either a constant value or increasing regularly with the molecular weight. The following table gives the results:—

	$K_t =$
For methyl chloracetate	$0{\cdot}3747 + 0{\cdot}00038t$
„ ethyl „	$0{\cdot}3900 + 0{\cdot}00038t$
„ propyl „	$0{\cdot}4067 + 0{\cdot}00038t$
„ allyl „	$0{\cdot}3888 + 0{\cdot}00038t$
„ methyl dichloracetate	$0{\cdot}3032 + 0{\cdot}00038t$
„ ethyl „	$0{\cdot}3215 + 0{\cdot}00038t$
„ propyl „	$0{\cdot}3335 + 0{\cdot}00038t$
„ allyl „	$0{\cdot}3244 + 0{\cdot}00038t$
„ methyl trichloracetate	$0{\cdot}2592 + 0{\cdot}00038t$
„ ethyl „	$0{\cdot}2778 + 0{\cdot}00038t$
„ propyl „	$0{\cdot}2892 + 0{\cdot}00038t$
„ allyl „	$0{\cdot}2806 + 0{\cdot}00038t$
„ allyl acetate and isobutyrate..	$0{\cdot}4305 + 0{\cdot}00088t$
„ allyl propionate, butyrate and valerate	$0{\cdot}4330 + 0{\cdot}00088t$
„ allyl benzoate	$0{\cdot}3732 + 0{\cdot}00075t$
„ ethyl and propyl oxalate	$0{\cdot}4199 + 0{\cdot}00066t$
„ „ „ malonate ...	
„ allyl oxalate.....	$0{\cdot}4122 + 0{\cdot}00066t$
„ isobutyl oxalate	$0{\cdot}4274 + 0{\cdot}00066t$
„ isoamyl „	$0{\cdot}4354 + 0{\cdot}00066t$
„ ethyl and propyl succinate ...	$0{\cdot}4391 + 0{\cdot}00066t$

	$H_t =$
For allyl succinate.....	0·4323 + 0·00066t
„ isobutyl succinate	0·4421 + 0·00066t
„ isoamyl „	0·4493 + 0·00066t
„ aniline and orthotoluidine....	0·4706 + 0·00070t
„ dimethylaniline	0·4018 + 0·00092t
„ diethyaniline	0·4343 + 0·00092t
„ chlorbenzene	0·2988 + 0·00074t
„ chlortoluene	0·3160 + 0·00074t
„ benzyl chloride	0·3225 + 0·00074t.

H. C.

Molecular Heats of Gaseous Substances. By H. Le Chatelier (*Zeit. physikal. Chem.*, **1**, 456—458).—The author finds from the determinations of Wiedemann, that the specific heats of gaseous substances may be represented within the limits of experimental error by the equation $C = 6{\cdot}8 + \alpha(273 + t)$, for temperatures below 200°, where C is the true specific heat at constant pressure, and α a coefficient depending on the nature of the gas, and having a higher value the more condensed the molecule. From this it would follow that as the temperature approaches the absolute zero, the molecular heats of gases and vapours would more and more approximate to the same limiting value, but in how far this may be considered a true law the experimental data are insufficient to determine. From the author's own experiments, he finds that the relation holds good for carbonic anhydride at 2000°, but for water-vapour at 3300° it no longer holds, probably owing to partial dissociation having already occurred.

H. C.

Air Thermometer. By L. Cailletet (*Compt. rend.*, **106**, 1055—1057).—A description of a modified form of air thermometer at constant volume, the gas employed being hydrogen. The upper part of the manometer tube is vacuous, and hence the correction for atmospheric pressure is eliminated. The pressure is adjusted by means of a movable reservoir of mercury, which can be cut off from the manometer tube by a screw tap. The point of constant volume is indicated by a platinum wire, which is connected with an electric current in such a way that when the mercury in the manometer touches the wire the circuit is completed, and a bell rings. The space between the bulb and the zero point, which contains hydrogen at an unknown temperature is only 0·006 of the volume of the bulb. The hydrogen thermometer and the mercurial thermometer agree between −30° and +50°.

C. H. B.

Air Thermometers. By Crafts (*Compt. rend.*, **106**, 1222—1225).—The author, like Cailletet (preceding Abstract), employs electrical connections as a means of adjusting the mercurial column in the manometer to zero, and he also makes use of an electrical stopcock, which cuts off the mercury as soon as the latter touches the platinum wire which marks the zero point. In accurate work, the whole of the mercury in the manometer and the stem of the thermometer should be immersed in melting ice.

C. H. B.

Latent Heat of Vaporisation of Volatile Substances. By J. CHAPPUIS (*Compt. rend.*, **106**, 1007—1008).—Cailletet and Mathias's determinations of the density of sulphurous anhydride (*J. de Phys.* [2], **6**, 414), together with Regnault's determinations of the maximum tensions of this substance between −30° and +65°, make it possible to calculate the latent heat of vaporisation of the liquid by means of the well-known formula, and the value thus obtained, 91·2 Cal. at 0°, agrees with the author's direct determination, 91·7 Cal. (Abstr., 1887, 627).

In the case of carbonic anhydride, direct experiment gives 56·25 Cal. at 0°. The value calculated from Sarrau's data is 55·95 Cal.; from the curves given by Cailletet and Mathias (*J. de Phys.* [2], **5**, 549), 57·48; and from their equation for the variation of the latent heat of carbonic anhydride with the temperature, 56·75 Cal. C. H. B.

Measurement of the Latent Heat of Vaporisation of Liquefied Gases. By E. MATHIAS (*Compt. rend.*, **106**, 1146—1148).—The liquefied gas is contained in a copper receiver gilded externally, from which it escapes through a serpentine metal tube of narrow diameter, the whole being placed in a Berthelot's calorimeter. During the evaporation of the liquid, which is regulated by means of stopcocks, the temperature of the water is kept within 0·05° of the initial temperature by the gradual addition of sulphuric acid of known strength, care being taken to ensure rapid and perfect admixture. The decrease in the weight of the metal receiver gives the quantity of liquid which has evaporated, and the amount of acid added gives the quantity of heat required to keep the water at the initial temperature, or, in other words, the quantity of heat absorbed by the volatilisation of the liquefied gas.

Experiments with sulphurous anhydride show that the latent heat of vaporisation diminishes as the temperature rises. C. H. B.

Calorimetry at Constant Temperature. By A. D'ARSONVAL (*Compt. rend.*, **106**, 1225—1226).—The author has for some time utilised the evaporation of ethyl chloride to compensate for the heat developed by the animals in thermo-physiological experiments. The water in the calorimeter is thus kept at the initial temperature, and the quantity of heat developed is calculated from the amount of ethyl chloride volatilised. (Compare Mathias, preceding Abstract.)

C. H. B.

Heat of Formation of Aniline. By P. PETIT (*Compt. rend.*, **105**, 1087—1089).—Nitrobenzene is completely converted into aniline in a few minutes by the action of chromous oxide at the ordinary temperature. This reaction was carried out in the calorimeter by mixing nitrobenzene with a measured quantity of a standard solution of chromous chloride containing free hydrochloric acid. The heat of oxidation of chromous chloride has been determined by Recoura, and the heat of formation of aniline hydrochloride by Berthelot. C_6 diamond + H_7 gas + N gas = C_6H_7N liquid absorbs −12·4 Cal. Aniline was also burnt in oxygen in the calorimetric bomb. Only a very small quantity of nitrogen oxides is formed. The heat of combustion for

1 gram is 8·794 Cal.; for a gram-molecule at constant volume 817·8 Cal., at constant pressure 818·5 Cal. C_6 diamond + H_7 gas + N gas = C_6H_7N liquid absorbs −13·0 Cal.

The two values agree well. C. H. B.

Thermochemistry of Diazo-derivatives. By L. Vignon (*Compt. rend.*, **106**, 1162—1165).—The determinations were made by taking advantage of the fact that in dilute solutions the formation of the diazo-derivatives is complete, and is not complicated by secondary reactions. Moreover, if the temperature is below 10°, some time elapses before the decomposition of the diazo-derivative becomes appreciable. Dilute solutions of hydrochloric acid, aniline, or orthotoluidine, or paratoluidine, and pure sodium nitrite were mixed in the calorimeter, and the following results were obtained:—

Diazobenzene chloride	+31·4 Cal.
Orthodiazotoluene chloride	+33·6 „
Paradiazotoluene chloride	+33·1 „

The heats of formation of aniline and toluidine hydrochlorides not yet having been determined, the heats of formation of the diazo-derivatives from their elements cannot be accurately calculated, but if the heat of neutralisation of nitrous acid by soda is taken as equal to the heat of neutralisation by baryta, the following values are obtained:—

Diazobenzene chloride	A − 44·0 Cal.
Orthodiazotoluene chloride	B − 41·8 „
Paradiazotoluene chloride	C − 42·3 „

Where A, B, and C, are the heats of formation of the hydrochlorides of aniline, orthotoluidine and paratoluidine, respectively. The formation of the diazo-derivatives themselves is undoubtedly endothermic, a result which explains their instability and the necessity for cooling the reagents during their formation. The development of heat which is observed is due to the formation of water and sodium chloride.

C. H. B.

Heat of Combustion of Coals from the North of France. By Scheurer-Kestner (*Compt. rend.*, **106**, 1092—1094, 1160—1161, 1230—1231).—The coal, in small fragments, was burnt in a rapid current of oxygen in a Favre and Silberman's calorimeter. Under these conditions, combustion is much more complete than if the coal is finely powdered. Twenty-one samples of coal were analysed, and their heats of combustion determined. The numbers actually obtained never agree with the values calculated from the composition or by means of Dulong or Cornut's equation. Usually the heat of combustion is greater than the sum of the heats of combustion of the constituents, but occasionally it is less. Cornut's formula gives the closest approximation to the real value. No explanation is yet possible of the difference between the real and calculated values. The important fact is that coal has been formed partly, at least, by endothermic reactions.

A coal from Gilly which contained 5 per cent. of oxygen gave a heat of combustion of 8553 Cal., whilst another coal from Sart-les-Moulins, containing only 1·5 per cent. of oxygen, developed only 8435 Cal. The heats of combustion of coals from the basin of North France vary between 8340 and 9257 Cal., the value usually lying between 8400 and 8800 Cal. It follows that these coals are somewhat richer than those of Saarbruck, which give numbers between 8215 and 8724. The highest results yet obtained were with two coals from Creuzot, namely, 9456 Cal. and 9623 Cal. C. H. B.

Expansion of Fluids and Change of Substance from the Fluid to the Gaseous State. By A. NADESCHIDIN (*Chem. Centr.*, 1888, 168, from *Rep. Phys.*, **23**, 617—649, and 685—718).—The different theories which deduce one property of liquids from the others, attach special importance to the determination of the critical condition. In the present memoir the results of the determination of the critical pressure, the critical temperature, and the critical volume are given.

Scarcely any determinations of the critical volume have previously been made, but it is necessary to know it in order to precisely estimate the critical temperature and critical pressure; for only when the space which contains the liquids and vapours is equal to the critical volume, does the substance attain the critical condition. The critical volume is obtained when the liquid only changes its volume up to a point at which the limiting surface between the liquid and vapour disappears.

This was determined for a series of compounds by means of a series of small tubes, a fraction of whose volume was filled with the desired liquid. Those tubes were chosen in which when heated all the fluid volatilised, and in which too the disappearance and reappearance of the meniscus on cooling occurred in the middle, and not in proximity to the end of the tube. A series of theoretical and experimental researches have been made on the expansion of liquids at equal temperatures and at temperatures lying between wide limits.

J. P. L.

Determination of the Specific Weight and of the Vapour-pressure of Mixtures of Sulphurous and Carbonic Anhydrides. By A. BLÜMCKE (*Ann. Phys. Chem.* [2], **34**, 10—21).—The experiments were carried out with apparatus of the same kind as that used by the author in his previous researches (Abstr., 1885, 215; 1887, 435). The liquids used for comparison with carbonic anhydride were ether, benzene, and light petroleum, and for comparison with sulphurous anhydride, and with mixtures of the two, the author used mixtures of chloroform and ethylic alcohol. In his former researches, the values obtained for the specific weight of carbonic anhydride differed considerably at the higher temperatures from those given by Cailletet and Mathias (*J. de Phys.* [2], **5**, 549). This he attributes to impurity in the carbonic anhydride used by him, and to his not having provided means of properly controlling the pressure at which the specific weight was determined, a defect which he has remedied in

the present research, and his results are now in close agreement with those obtained by these physicists.

The degree of concentration of the mixtures was not found to undergo alteration when the temperature was increased; for the author observed no sign of the escape of bubbles of carbonic anhydride, or of the formation of an emulsion, which latter was stated by Pictet (*Nouvelles machines frigorifiques*, 1885, 11) to occur when cooled below zero.

By a separate series of experiments, the author found that the percentage composition could be determined to a high degree of approximation from observations of the pressure.

The vapour-pressure was found to depend on the amount of fluid introduced into the apparatus, showing that the vapour was not saturated. This had previously been observed by Linde (*Allg. Hop. Brauerzeit.*, 1888, 299).

The specific weight was determined from the following interpolation formula:—

$$V(1 + n\alpha) = \frac{Vs + n\sigma}{S},$$

where p is the percentage by weight of carbonic anhydride in the mixture, n the number of volumes, at a pressure and temperature of 760 mm. and 0° respectively, of gaseous carbonic anhydride to an equal volume of liquid sulphurous anhydride, V the original volume of sulphurous anhydride, s its specific weight, $\sigma = 0{\cdot}001965$, the specific weight of gaseous carbonic anhydride at 0° and 760 mm., S the specific weight of the mixture, and α a coefficient which, as in the case of mixtures of alcohol and carbonic anhydride, was not a constant, but increased with the value of p and with the temperature. With regard to the vapour-pressure, it was impossible for the author to compare his results numerically with those obtained by other experimenters, owing to the vapour being unsaturated, as in no case had the volume been determined. The principal question to be determined was therefore whether the vapour-pressure of sulphurous anhydride could at any temperature be diminished by the addition of carbonic anhydride, or whether its boiling point could be raised in the same manner.

According to Pictet (*Arch. Gen.*, **13**, 212) and Corsepius, the so-called Pictet fluid has a vapour-pressure, at temperatures above 30°, which is less than that of pure sulphurous acid, and, according to Grätz (*Allg. Brauer Hopfenzeit.*, **17**, 1157) Turrettini and Rossi have obtained similar results. Rilliet obtains diametrically opposite results, and the author's observations confirm those of Rilliet. The author was unable to obtain any mixture with a higher boiling point than that of pure sulphurous anhydride, whilst Pictet, for example, gives $-7{\cdot}5°$ for the boiling point of the mixture $CO_{16}S_7$, the author finds that at $-17°$ it would have a pressure of 4 atmos.

G. W. T.

Internal Friction of Liquids. By L. Graetz (*Ann. Phys. Chem.* [2], **34**, 25—39).—The author observes that the large number of researches which have been made, from those of Poiseuille down-

wards, have not led to the determination of any definite relations between the internal friction of liquids and their chemical constitution. Graham was led to believe that such a relation must exist from the well-defined maximum values of internal friction in solutions of hydroxides.

With the object of finding such a relation, a large number of organic liquids were investigated by Graham himself (*Annalen*, **123**, 90), and also by Rellstab ("Transpiration homologer Flüssigkeiten," *Inaug. Diss.*, Bonn, 1868), Guerout (*Compt. rend.*, **81**, 1027), and Pribram and Handel (*Wien. Ber.*, **78**, 113; **80**, 1; **84**, 11), and a large number of saline solutions were experimented on by others. The results with the latter were practically *nil*, and those obtained with the former did not lead to the discovery of any definite relations, although the differences were found to be much greater than in gases, and the relations which appeared to hold in some cases were subject to numerous exceptions. The author formerly (*Ann. Phys. Chem.* [2], **25**, 340) came to the conclusion that the internal friction is not due mainly, as in gases, to molecular impacts, but to the direct action of molecular forces, he therefore considers only the residual internal friction after subtracting that of the substance in a gaseous state, the latter being due to molecular impacts.

Maxwell (*Phil. Mag.* [4], **35**, 129) showed that if μ is the coefficient of internal friction, E the modulus of rigidity, and T what he calls the *time of relaxation*, namely, the time during which a stress excited in the liquid falls to $1/e$ of its original value, then $\mu = ET$, independently of any hypotheses with regard to the nature of the internal friction. Starting with this equation, the author shows that to a first approximation, $\mu = A(\theta_0 - \theta)/\theta$, where θ is the temperature of the fluid, and θ_0 its critical temperature, both reckoned from that temperature for which μ becomes infinite. Calling this unknown temperature t_1, the formula in centigrade degrees becomes

$$\mu = A(t_0 - t)/(t - t_1),$$

in which A and t_1 are unknown constants which have to be determined for each liquid.

The author made experiments on 61 different liquids, for which the critical temperature was either already known or could be determined, and in 54 of these he found that the observations were in close agreement with the formula. The seven exceptions consisted of ethylic ether and the six alcohols included in the liquids investigated. Those which agreed with the formula included various fatty acids, ethereal salts, haloïd compounds of the fatty series, aldehydes, ketones, aromatic hydrocarbons, and water.

The author then calculated the values of a quantity $G = A/N$, where N is the number of molecules in unit volume of the liquid. The quantity G is a measure of the viscosity which is comparable for different liquids.

If n is the number of molecules per unit of length in the liquid (Hagenbach, *Ann. Phys. Chem.*, **109**, 358), the author gives the following table of the values of G for the normal acids and ethereal salts of these acids for values of n from 1 to 4.

	Acids.	Methyl salts.	Ethyl salts.	Normal propyl salts.	Normal butyl salts.	Isopropyl salts.	Isobutyl salts.
$n = 1$	747·7	—	942·3	1246·2	1725·2	1519·8	1593·0
$n = 2$	1039·0	1080·4	1246·8	1401·5	2034·5	1632·5	1802·2
$n = 3$	1320·5	1277·2	1539·2	1616·5	—	1993·6	2090·0
$n = 4$	1578·7	1506·5	1702·5	1780·0	—	2150·2	—

If each horizontal line be subtracted from the one above it, we get the following differences for each CH_2-group in the acids—

	Acids.	Methyl salts.	Ethyl salts.	Normal propyl salts.	Normal butyl salts.	Isopropyl salts.	Isobutyl salts.
	291·3	—	304·5	155	309	236	209
	281·5	196·8	292·4	215	—	236	287
	258·2	229·3	163·3	164	—	156	—
Means	277·0	213·0	260·8	185	309	210	248

The differences of these mean values are not very great, and are within the limits of errors of observation. Similar relations are obtained in the case of organic chlorine and bromine compounds.

G. W T.

Osmotic Pressure in the Analogy between Solutions and Gases. By J. H. VAN'T HOFF (*Zeit. physikal. Chem.*, 481—508).—The object of this paper is to establish an analogy which is supposed to exist between the state of substances in dilute solution and the same in the gaseous condition, and to show that osmotic pressure in the former is comparable with vapour-tension pressure in the latter case.

Suppose a solution contained in a vessel of semi-permeable material, that is to say, material permeable only by water molecules but not by the molecules of the dissolved substance, and that this vessel is immersed in water. Then, owing to the attraction of the molecules of the dissolved substance, water will enter the vessel, thus causing an increase of pressure, and this will continue until the pressure within the vessel is sufficient to counterbalance the attraction exercised by the molecules of the dissolved substance. Equilibrium being thus established, the pressure within the vessel is equal to the osmotic pressure, and may be taken as a measure of the same.

If we suppose the vessel to be fitted with a movable piston as a means of altering the pressure at will, it will then be possible to produce any alterations in the concentration and allow water to either enter or leave the vessel by decreasing or increasing the pressure. In this way a cycle of reversible changes may be performed, such as

decreasing the pressure and allowing water to enter, and then by restoring the pressure bringing the solution back to its original state, and the sum of the work done in such changes being zero, the second law of thermodynamics may be applied.

Vessels of such semi-permeable materials are the cells of plants, and observations with these have shown by the comparison of isotonic solutions (solutions of equal osmotic pressure) that the pressure is directly proportional to the concentration. Also if a porous cell, the walls of which have been thoroughly impregnated under pressure with copper sulphate solution to drive out the air, be filled with copper sulphate solution and then immersed in a solution of ferrocyanide, the two will by diffusion meet in the walls of the cell and there form a layer of copper ferrocyanide, which will act as a semi-permeable material to cane-sugar solution. Observations made by Pfeffer on cane-sugar solution with the aid of such a cell have shown that up to a 5 per cent. solution the pressure is directly proportional to the concentration. But this proportionality between pressure and concentration is precisely similar to Boyle's law in the case of gases, and if we consider the entrance of the water and subsequent increase of pressure to be in proportion to the number of molecules which come into contact with the walls of the cell in a given space of time, the argument is similar to that by which Boyle's law is looked on as due to molecular bombardment, and leads of course to the same conclusions.

By the aid of one of the reversible changes before mentioned, it may be shown that Gay-Lussac's law should also hold for solutions, and that osmotic pressure should be proportional to the absolute temperature. The experiments of Pfeffer with cane-sugar and sodium tartrate solution give numbers which are in satisfactory agreement with this law, as also the numbers obtained from observations of isotonic solutions. The observations of Soret, who kept two parts of the same solution at different temperatures and then determined the strength of each, also give numbers agreeing with the above.

It may be shown by means of reversible cycles that the osmotic pressure of a gas when in solution is equal to the tension of the same gas when in the free state. This of course leads directly to Avogadro's law in solution, so that for equal osmotic pressures and equal temperatures equal volumes of different solutions contain the same number of molecules, namely, that which is contained in the same volume of a gas under the same conditions of pressure and temperature. That this relation actually holds in practice is shown by a comparison of the osmotic pressures of a 1 per cent. sugar solution at different temperatures with the calculated tension of hydrogen gas containing the same number of molecules at the same temperatures.

If in the general expression for the relation of pressure, volume, and temperature in gases $PV = RT$, in accordance with Horstmann's suggestion, kilogram-molecules be always taken, the value of R becomes constant for all gases. The value of this constant is 845·05. The author points out that this number is in direct numerical relation with that which expresses in calories the value of a kilogram-metre, or the work equivalent $A = 1/423$. Hence $AR = 2$ approximately,

and the simple relation is obtained APV = 2T, a relation which may prove of value in future thermodynamical work.

From the theorem that solutions in the same solvent which have equal maximum vapour-tension or freezing point, must be isotonic, which is easily proved by the aid of a reversible cycle, Raoult's laws of the molecular reduction of vapour-tension and freezing point may be deduced, on the assumption that Avogadro's law holds good for solutions. The numbers thus calculated theoretically are in excellent agreement with those observed, and thus afford additional indirect support to Avogadro's law in solution. The molecular reduction of the freezing point t is found to be equal to $0{\cdot}02\ T^2/W$, where T is the freezing point in absolute temperature and W the latent heat of fusion of the solvent. In the case of ethylene bromide, the latent heat of fusion of which was unknown, the number calculated from the known molecular reduction of the freezing point was 13, and a subsequent determination by Pettersson gave as the observed value 12·94.

The marked exceptions which occur to Raoult's laws, especially in the case of electrolytes, find an explanation in the assumption first made by Arrhenius, that these latter in solution are partially dissociated into their ions. This must of course lead to a modification of Avogadro's law. This may be expressed in the general formula APV = 2T, on the assumption that as dissociation of the molecules occurs these have i times their original numerical value, by the relation APV = $2i$T. The value of i is easily calculated from Raoult's experiments, for since the molecular reduction of the freezing point in aqueous solutions of cane-sugar, which strictly obey Avogadro's law, is 18·5 and in this case $i = 1$, in other cases i will simply be the observed molecular reduction divided by 18·5.

A second method of calculating the value of i is given by Guldberg and Waage's theory, the application of which to the present case is shown in the paper. The agreement of the numbers thus obtained by different methods is a sufficiently satisfactory one, and therefore in its altered form Avogadro's law may be still said to hold good for dilute solutions. H. C.

Chemical Equilibrium in Dilute Solutions. By M. Planck (*Ann. Phys. Chem.* [2], **34**, 139—154).—In a previous memoir (*Ann. Phys. Chem.* [2], **32**, 462), the author showed that the observed phenomena respecting the freezing and evaporation of dilute solutions could only be brought into accordance with the laws of thermodynamics by assuming that the molecules of the substance in solution undergo chemical decomposition to a greater or less extent, and found that van't Hoff's coefficient i (preceding Abstract) must be proportional to this chemical decomposition, or in other words must express the ratio of the actual number of molecules of the substance in solution to the number which there would be if no chemical decomposition took place. Arrhenius (*Zeit. physikal. Chem.*, **1**, 631) arrives at a similar conclusion, founded on the analogy between osmotic pressure in dilute solutions and the pressure of perfect gases. This analogy receives a good deal of support from chemical considerations inde-

pendently of purely theoretical ones, and the author thinks that it will probably be found to be in agreement with the theory derived from thermodynamical considerations; it will therefore be of interest to follow it into some of its consequences. The condition of equilibrium in a dilute solution must then be of the same character as the chemical equilibrium of various mutually reacting substances. The constituents of the molecules must themselves be considered as acting on each other, and not be treated as a mere mechanical mixture. If, for example, a dilute solution of hydrogen chloride, for which i is very nearly equal to 2, consisted of a mixture of free atoms of hydrogen and chlorine, these atoms could be either mixed or separated without expenditure of work, which is contrary to experience.

Let $\theta_0 - \theta$ be the lowering of the freezing point, $p_0 - p$ that of the pressure, θ_0 and p_0 being the temperature and pressure of the pure solvent, n_0 the number of molecules in the solvent, q_0 the heat of fusion of one of them, then, as the author has shown in the memoir previously referred to, n, the number of molecules of the substance in solution, is given by the equation—

$$n = \frac{n_0 q_0}{\theta_0^2}(\theta_0 - \theta) = n_0 \frac{p_0 - p}{p_0}.$$

Calling N the value which n would have if there were no chemical changes, n is generally greater than N for aqueous solutions, but is often less than N for benzene solutions. The value of i is n/N. Some of the molecules of the substance may enter into combination with molecules of the solvent, as occurs in the formation of hydrates, but as this does not alter the number of molecules in the solution, it cannot be determined from observations of the freezing point or of the pressure.

When a molecule can only break up in one way into two constituents, as is often the case for aqueous solutions, then if n_1 is the number of undecomposed molecules, and n_2 the number broken up into two constituents, $\mathrm{N} = n_1 + n_2$, and $n = n_1 + 2n_2$, so that we have—

$$i = \frac{n}{\mathrm{N}} = \frac{n_1 + 2n_2}{n_1 + n_2} = \frac{n_0}{\mathrm{N}} \cdot \frac{q_0(\theta_0 - \theta)}{\theta_0^2} = \frac{n_0}{\mathrm{N}} \cdot \frac{p_0 - p}{p_0},$$

n_1 and n_2 can therefore be uniquely determined in terms of N and i. The author confines himself to the consideration of solutions of this character. He finds that i depends on the temperature and the pressure, and also on the normal concentration N/n_0.

Where K is a quantity depending on the pressure and temperature only, M is the normal molecular weight of the substance in solution, and $\mathrm{K}/100\mathrm{M} = k$, the author finds that the law connecting the molecular freezing point and lowering of vapour-pressure for dilute aqueous solutions is given by the equation—

$$i = 1 + \frac{\sqrt{1 + 2k\mathrm{P}} - 1}{k\mathrm{P}}.$$

Thence it is only in the limiting cases $k = \infty$ and $k = 0$ that the

ratio of the fall in the freezing point to the percentage strength of the solution is constant (Blagden's law), or the fall in the vapour-pressure in a constant ratio to the percentage strength (Wüllner's law), and in these cases there is either no dissociation or it is complete. Equations of a very similar character are obtained for other solvents.

Tamman's researches (*Mém. Acad. St. Pétersbourg* [7], **35**, No. 9) on the vapour-pressure of aqueous solutions of various degrees of concentration, offer a means of testing the results obtained theoretically from the above equation. The results, however, do not agree with the theoretical ones, as in most of the experiments i increased with the concentration instead of diminishing. The author, however, points out that in the gases which are in a state of partial dissociation, it sometimes takes months before equilibrium is established: according to the analogy assumed to exist between gases and dilute solutions, a similar phenomenon may take place in the solutions, and this would seem to explain the discrepancies, although it would make it impossible to arrive at any definite results from measurements of the freezing point and vapour-pressure. Arrhenius, however, has shown (*loc. cit.*) that the value of i may be determined from the value of the electrical conductivity of the liquid, for if α is the coefficient of activity of the solution, that is, the ratio of the actual molecular conductivity to its value for an infinitely dilute solution, then for binary electrolytes, which are the only ones considered in the paper, $i = 1 + \alpha$. This gives a definite value for i, as the measurements of conductivity can be carried out very accurately, and are found to be in close agreement with each other. This leads to the inference that the establishment of chemical equilibrium in dilute solution is greatly facilitated by the passage through it of alternate currents, which calls to mind the assistance to the establishment of physical equilibrium rendered by mechanical disturbance.

The author finds that the conductivity, K, of a dilute solution is given by the equation—

$$K = \text{constant} \,.\, (i - 1)\, P = a(\sqrt{1 + bP} - 1),$$

where P is the percentage (either by weight or volume, the equation being only a first approximation) of dissolved substance, and a and b are functions of the pressure and temperature only.

This formula agrees substantially with one given by Ostwald (*Zeit. physikal. Chem.*, **2**, 36). It gives much better results than the ordinary quadratic interpolation formula, but the author finds that the theoretical formula obtained by proceeding to the second approximation, although too complicated to be convenient, gives results which are in close agreement with those obtained experimentally. Even this formula, however, gives no explanation of the existence of a maximum value of the conductivity, as is observed, for example, in the case of lithium chloride. The theory cannot, therefore, be applicable to concentrated solutions. G. W. T.

Laws of Chemical Equilibrium. By H. Le Chatelier (*Compt. rend.*, **106**, 1008—1011).—A continuation of the discussion with Duhem.

Liebrich's Inactive Space. By R. Gartenmeister (*Annalen*, **245**, 230—235).—The author has repeated Liebrich's experiments on the existence of an inactive zone ("todten Raum") surrounding the space in which chemical reaction takes place in liquids. He finds that the hypothesis of this inactive zone is unnecessary, as the phenomena in question can be easily explained without its aid. For example, a test-tube is nearly filled with a mixture of sodium carbonate and chloral hydrate solution. It is then closed with a cork and inverted; after five minutes have elapsed the mixture becomes cloudy (owing to the formation of chloroform) except at the upper surface of the liquid. This remains clear, because the chloroform evaporates into the space above the surface of the liquid. When this space is saturated with chloroform vapour, then chloroform begins to separate out in the uppermost layers of the mixture. W. C. W.

Formation of Layers in Mixtures of Alcohol, Water, and Salts, or Bases. By J. Traube and O. Neuberg (*Zeit. physikal. Chem.*, **1**, 509—515).—Bodländer was the first to observe that on dissolving ammonium sulphate in mixtures of alcohol and water, at certain concentrations, the liquid divides into two well-defined layers. The authors find a similar behaviour with potassium and sodium hydroxides and carbonates, sodium phosphate and sulphate, zinc and magnesium sulphates, and other salts. They have therefore examined this change in the case of ammonium sulphate under varying conditions of temperature and concentration.

With a solution containing 340 grams of salt per litre, 750 c.c. of which is mixed with 250 c.c. of alcohol (99·6 per cent.), it is found that with increasing temperature there is in the upper layer a decrease in the relative amounts of water and salt, and an increase in that of the alcohol; in the lower layer, there is an increase of water, but a decrease of salt and alcohol. The change in the composition of the lower layer is, however, so small, that within tolerably wide limits of temperature it may be looked on as constant. Keeping the temperature constant, and increasing either the amount of alcohol or salt in solution, it is found that in the upper layer there is a decrease in the relative amounts of water and salt and an increase in that of the alcohol, in the lower layer there is a decrease in the alcohol and an increase in the salt, the water first increasing and then decreasing. In this way the addition of 40 grams of salt to a litre produce about the same effect as the addition of 100 grams of alcohol.

Experiments with potassium carbonate led to similar conclusions as those above quoted. It was not possible in either case to determine whether the components of the layers are present in definite molecular proportions, but this appears likely, especially in the case of the lower layer, the percentage composition of which has a great tendency to remain constant. H. C.

Absorption of Gases by Grey Vulcanised Caoutchouc. By G. Hüfner (*Ann. Phys. Chem.* [2], **34**, 1—10).—As the result of a series of observations extending over a considerable period, the author arrives at the following conclusions.

Within the limits of temperature from 5° to 25°, no definite absorption coefficient of atmospheric air can be determined. An apparent absorption does take place, and during a series of experiments extending over six months this absorption was found to continue at a nearly uniform rate, but the author finds that it is due to a gradual disappearance of oxygen, caused probably by slow oxidation of the caoutchouc.

Within the temperature limits from 15° to 25°, there is no perceptible absorption of nitrogen. At a temperature of about −2° grey vulcanised caoutchouc absorbs about its own volume of dry carbonic anhydride. The specific coefficient of absorption of this gas diminishes with increasing temperature.

Within the limits of temperature from −2° to −13°, there was found to be no perceptible absorption of hydrogen, even after the caoutchouc had been in contact with the gas for three months.

G. W. T.

Inorganic Chemistry.

Methods for obtaining Constant Streams of Hydrogen Chloride, Ammonia, and Nitrogen. By G. Næumann (*J. pr. Chem.* [2], **37**, 342—345; compare Abstr., 1887, 769).—Hydrogen chloride can be generated in a Kipp's apparatus by the action of ordinary sulphuric acid on carnallite, and ammonia gas by allowing a solution of ammonia to react with solid potassium hydroxide. When the materials are exhausted, the solution of potash which is formed may be used for ordinary laboratory purposes after it has been boiled to expel ammonia.

To prepare nitrogen in a Kipp's apparatus, it is best to employ cubes containing chloride of lime made according to Winkler's method (Abstr., 1887, 442); these are treated with a mixture of equal volumes of ammonia and water. The resulting gas contains suspended ammonium chloride and other impurities, which may be removed by passing it through water, potash, and sulphuric acid.

The author describes an apparatus for obtaining constant supplies of these and other gases, which is superior to Kipp's, in that it is cheaper and that broken parts can be easily replaced. G. T. M.

Hyposulphates. By K. Klüss (*Chem. Centr.*, 1888, 215—216).—The following hyposulphates (dithionates) are described:—Bismuth, $Bi_2O_3,S_2O_5 + 5H_2O$; stannous, $8SnO,S_2O_5 + 9H_2O$; thorium, $Th(S_2O_6)_2 + 4H_2O$; normal and basic chromium salts, $Cr_2(S_2O_6)_3 18H_2O$ and $3Cr_2O_3,4S_2O_5 + 24H_2O$; uranium salts of the composition $7UO_2,S_2O_5 + 8H_2O$, $6UO_2,S_2O_5 + 10H_2O$, and $8UO_2,S_2O_5 + 21H_2O$; an ammonium salt, $(NH_4)_2,S_2O_6,\frac{1}{2}H_2O$; a beryllium salt, $5BeO,2S_2O_5 + 14H_2O$; ferrous hyposulphate, $FeS_2O_6 + 7H_2O$, and the following basic ferric salts, $8Fe_2O_3,S_2O_5 + 14H_2O$, and $3Fe_2O_3,S_2O_5 + 8H_2O$. The normal ferric

salt is unstable in the solid state. Normal copper hyposulphate, $CuS_2O_6 + 5H_2O$, and several basic salts, $4CuO,S_2O_5 + 3$ and $4H_2O$, a mercury salt, $5HgO,2S_2O_5$, and an aluminium salt, $Al_2O_3,3S_2O_5+18H_2O$, were also prepared. Attempts to prepare hyposulphate of zirconium and vanadium failed. Hyposulphuric acid is without effect on cerium dihydroxide and thallium hydroxide at the ordinary temperature; on heating, the acid decomposes with evolution of sulphur dioxide, which reduces the hydroxide.

In the second part of the paper, the author discusses the isomorphism of the hyposulphates, and describes the preparation of different double salts. Double salts of ammonium hyposulphate with hyposulphates of magnesium, zinc, cadmium, ferrous oxide; manganous oxide, nickel, cobalt, aluminium, and copper are described. The following isomorphous mixtures were analysed: barium with lithium and silver salts, and thallium with sodium, lithium, silver, and barium salts. There does not appear always to be such a sharply defined line between double salts and an isomorphous mixture as has hitherto been supposed. J. P. L.

Nitrous Anhydride and Nitrosyl Chloride. By A. Geuther (*Annalen*, **245**, 97—99).—The mixture of nitrogen sesquioxide and peroxide obtained by the action of nitric acid (sp. gr. 1·4) on arsenious oxide, may easily be separated by distillation, if the vapours are first passed into a flask at 30° before entering the receiver, which is surrounded by ice and salt. The sesquioxide boils at 3·5°. Its specific gravity has been determined at different temperatures—

−8°	1·464	0°	1·449
−4	1·4555	+1	1·4485
−1	1·451	+2	1·447

The peroxide boils at 26°. Its specific gravity varies from 1·5035 at −5° to 1·4935 at 0°, and 1·474 at 15°.

The specific gravity of nitrosyl chloride is—

At	−18°	1·433
	−15	1·425
	−12	1·4165

Nitryl chloride does not appear to exist. W. C. W.

Action of Carbon Tetrachloride on Inorganic Oxygen Compounds free from Hydrogen. By H. Quantin (*Compt. rend.*, **106**, 1074—1076).—The various oxygen compounds were heated at different temperatures in the vapour of the tetrachloride.

Barium and sodium carbonates at dull redness are completely converted into chlorides with evolution of carbonic anhydride and carbon oxychloride. Geuther obtained a mixture of carbonate and chloride by the action of the tetrachloride on the oxides of these metals, but probably the chloride which was formed melted and protected the carbonate from the action of the vapour.

Carbonic anhydride, boric anhydride, and silicic anhydride offer

similar resistances to the reducing action of carbon tetrachloride, and hence it was of interest to compare the behaviour of carbonates, borates, and silicates. Ferric and aluminium borates are completely volatilised at a red heat, the boric acid being partially converted into boron trichloride and partially volatilised as the anhydride. Kaolin is distinctly attacked under the same conditions. Silicates rich in silica yield a small quantity of silicon tetrachloride, the amount increasing with the proportion of alumina in the silicate.

Barium and potassium sulphates at a red heat are partially converted into chlorides, with formation of pyrosulphuryl chloride, carbonic anhydride, and carbon oxychloride.

Calcium phosphate at a red heat yields calcium chloride and phosphorus pentachloride. By the action of chlorine and carbonic oxide on the phosphate at a lower temperature, Riban obtained phosphorus oxychloride. It follows that the interaction of carbon tetrachloride and phosphorus oxychloride at a red heat yields phosphorus pentachloride.

Tungstic anhydride at dull redness yields the oxychlorides $WOCl_2$ and $WOCl_4$, whilst molybdic and uranic anhydrides yield mixtures of chloride and oxychloride.

From these results it follows that oxygen compounds which contain no hydrogen are attacked by carbon tetrachloride at a high temperature, the metal and the non-metal being converted into oxychloride, if the latter can exist in presence of excess of carbon tetrachloride. The tetrachloride is converted into carbonic anhydride and carbon oxychloride. If the temperature is so high that the oxychlorides are reduced by the excess of the tetrachloride, metallic and non-metallic chlorides only are obtained. C. H. B.

Sodium Potassium Carbonate. By L. Hugounenq and J. Morel (*Compt. rend.*, **106**, 1158—1160).—A solution containing per litre 495·9 grams of potassium iodide, 10·3 grams of potassium carbonate, 77·3 grams of sodium carbonate, 42·0 grams of disodium phosphate, and 58·3 grams of sodium chloride, when allowed to evaporate spontaneously, yields large, transparent, prismatic crystals containing the two carbonates together with water of crystallisation. They are slightly efflorescent, begin to melt at about −40°, and dissolve in their own weight of water. The crystals are monoclinic prisms, the dominant face being me^1, and the ratios of the axes $a : b : c = 0{\cdot}7104 : 1{\cdot}0 : 0{\cdot}78$, with an inclination of 75° 35′. When dissolved in water and recrystallised, the crystals have not the same composition, but contain a higher proportion of potassium, and approximate more closely to Margueritte's salt, $K_2CO_3,6H_2O + 2(Na_2CO_3,6H_2O)$. The composition of the original crystals agrees fairly well with the formula $K_2CO_3,6H_2O + 3(Na_2CO_3,6H_2O)$, but only when they have been formed under the particular conditions described above. There can be little doubt that the crystals are not true double salts, but mixtures of isomorphous salts. C. H. B.

Compound of Zinc Oxide with Sodium Hydroxide. By A. M. Comey and C. L. Jackson (*Ber.*, **21**, 1589—1590).—When a solu-

tion of zinc or zinc oxide in strong aqueous soda is shaken with alcohol, the liquid separates into two layers. The lower, aqueous, layer is again treated two or three times with alcohol, when it solidifies to a crystalline mass. The alcoholic solution, under circumstances not yet determined, deposits lustrous needles in conical or spherical groups. The crystals have the composition expressed by the formula $2NaOZnOH + 7H_2O$. They do not melt at 300°, and are decomposed by alcohol, and by water when free alkali is not present. The composition of the crystals first mentioned was not determined, but they seem to differ from the other crystals only in having a larger amount of water of crystallisation; they melt below 100°.

N. H. M.

Action of Sodium Thiosulphate on Cupric Salts. By G. VORTMANN (*Monatsh.*, **9**, 165—179).—The compound obtained when a solution of sodium thiosulphate is added to a solution of copper sulphate has been investigated by many experimenters, and various formulæ have been assigned to it. The author's results are as follows. A greenish-yellow salt, $Cu_2S_2O_3,Na_2S_2O_3 + 3H_2O$, separates in the form of microscopic prisms when saturated solutions of copper sulphate and sodium thiosulphate in the proportion of 1 mol. of copper sulphate to 2 mols. of sodium thiosulphate are mixed and allowed to stand at the ordinary temperature. Sodium tetrathionate is also formed. When, however, the solutions are previously heated to 40° and then mixed, the temperature rises 5—7°, and on standing in water at 40° an intensely citron-yellow salt, $3Cu_2S_2O_3,2Na_2S_2O_3 + 8H_2O$, separates in the form of microscopic prisms. When this salt is washed with alcohol and dried over sulphuric acid, it loses 3 mols. H_2O, but if left too long it gradually decomposes. This citron-yellow salt can also be prepared from the greenish-yellow one by continued washing with cold water, and it is always formed when the solutions of copper sulphate and sodium thiosulphate are warm or dilute. It is unstable, decomposes, and becomes black on standing. When boiled with water or dilute sulphuric acid, it is decomposed: $3Cu_2S_2O_3,2Na_2S_2O_3,8H_2O = 3Cu_2S + 2Na_2SO_4 + H_2SO_4 + S_2 + 2SO_2 + 7H_2O$.

Attempts to prepare copper thiosulphate were unsuccessful.

Salts containing more than 1 mol. of sodium thiosulphate to 1 mol. of copper thiosulphate, such as $Cu_2S_2O_3,2Na_2S_2O_3 + 4H_2O$; $Cu_2S_2O_3,3Na_2S_2O_3 + 2H_2O$, and $Cu_2S_2O_3,4Na_2S_2O_3 + 6H_2O$, are best obtained by mixing a solution of the citron-yellow salt with a solution of the calculated quantity of sodium thiosulphate and adding alcohol. The double salts separate as oily liquids which solidify when treated with fresh quantities of absolute alcohol. They are yellow to white, crystalline compounds, which are more stable and less readily decomposed when boiled with water than the salts described above. They do not decompose on keeping, and are readily soluble in water, but are not deliquescent. With barium salts, their aqueous solutions give a white precipitate, $Cu_2S_2O_3,2BaS_2O_3 + 7H_2O$, which is soluble in hydrochloric acid, but almost insoluble in water.

F. S. K.

Vapour-density of Aluminium Chloride and the Valency of the Elements of the Aluminium Group. By L. F. NILSON and O. PETTERSSON (*Zeit. physikal. Chem.*, **1**, 459—464).—Aluminium chloride, when heated in an atmosphere of carbonic anhydride, gives at a temperature a little below 835°, a vapour-density agreeing with the formula $AlCl_3$, without any sign of decomposition. If heated above 935°, the chloride begins to attack the platinum vessel.

Since for indium chloride the formula $InCl_3$ has been established, there can be little doubt of the trivalency of the aluminium metals. It is extremely probable that in accordance with this view, gallium chloride has the formula $GaCl_3$, which, indeed, Friedel's experiments seem to indicate. H. C.

Passivity of Iron and Nickel. By E. SAINT-EDME (*Compt. rend.*, **106**, 1079—1080).—Commercial sheet nickel is passive in ordinary nitric acid, whereas iron is only passive in the fuming acid; but iron in contact with nickel becomes passive in the ordinary acid. Iron becomes passive gradually, but nickel is passive immediately. If iron and nickel are introduced into the acid together, the iron likewise becomes passive instantly. Electrolytic nickel from an ammoniacal solution of the chloride or sulphate is passive immediately.

Iron is only passive when azotised, and loses its passivity if heated in hydrogen. Passive nickel when heated to bright redness in a current of pure hydrogen yields a small quantity of ammonia and acquires a silvery lustre, but remains passive, and hence it would seem that the nickel retains some of the nitrogen very tenaciously. C. H. B.

Nickel Ammonium Oxalate. By K. KRAUT (*Annalen*, **245**, 239—240).—Nickel oxalate dissolves in ammonia, and the solution deposits green, microscopic crystals of the composition $2NH_3,NiC_2O_4 + 5H_2O$. The compound effloresces on exposure to the air and loses 3 mols. H_2O. W. C. W.

Phosphotungstic and Arsenotungstic Acids. By F. KEHRMANN (*Annalen*, **245**, 45—57).—The derivatives of phosphotungstic acid have been previously described by the author (Abstr., 1887, 777). The arsenotungstic acid which Fremery obtained by adding dilute sulphuric acid to barium tungstate suspended in a solution of arsenic acid, is not a homogeneous compound, but consists of a mixture of two acids. The separation of the acids is accomplished by the method used for the separation of the phosphotungstic acids (*loc. cit.*).

α-Arsenoluteotungstic acid, $AsH_3W_8O_{28} + 16H_2O$, is analogous to the phospholuteotungstic acid, and the salts of the two acids are isomorphous. The potassium salt, $K_3AsW_8O_{28} + 8H_2O$, is triclinic. The acid is very stable; the arsenic acid is not reduced by sulphuretted hydrogen. W. C. W.

Titanium. By T. KOENIG and O. v. D. PFORDTEN (*Ber.*, **21**, 1708—1716).—*Titanic acid trichloride*, $TiCl_3{\cdot}OH$, is readily obtained by mixing the theoretical quantities of titanium chloride and 36 per cent. hydrochloric acid, the reaction being extremely energetic. It is

a yellow, spongy, very deliquescent mass which dissolves very readily in water and alcohol with a hissing noise; it also dissolves in ether with a similar energetic reaction, but when more ether is added, a white, deliquescent compound is precipitated. An aqueous solution is tolerably stable, but titanic acid is precipitated after continued boiling. When heated in the dry state, it is decomposed into titanic acid and titanic chloride; the same decomposition takes place when it is heated in a current of hydrogen or hydrogen chloride.

Titanic acid dichloride, $Ti(OH)_2Cl_2$, is prepared by adding titanium chloride to a slight excess of hydrochloric acid; by the action of titanium chloride on the calculated quantity of 36 per cent. hydrochloric acid; or by adding titanium chloride to the requisite quantity of well-cooled water. It is a very voluminous, yellow, exceedingly deliquescent mass or a vitreous solid, according to the method of preparation. It is soluble in alcohol and ether, but a white compound is precipitated when an ethereal solution is strongly diluted with absolute ether. Titanic acid separates when an aqueous solution is boiled. When heated in the dry state, it is rapidly decomposed, yielding titanic acid and hydrogen chloride.

Titanic acid monochloride, $Ti(OH)_3Cl$, is formed when the dichloride or trichloride is allowed to remain for some time exposed to the air at the ordinary temperature. Both compounds deliquesce, with evolution of hydrogen chloride, but at the end of about a month's time they solidify. The product contains, approximately, 2 mols. H_2O after remaining for two months over sulphuric acid. When placed in a desiccator containing phosphoric anhydride, it loses 1 mol. H_2O in 14 days, and after a long time the anhydrous compound is obtained. In one experiment it was obtained in a crystalline form. It is a white compound which is stable in the air, and far less readily soluble in water than the dichloride or trichloride. In order to obtain a clear aqueous solution of the anhydrous compound, a few drops of hydrogen chloride must be added; titanic acid is precipitated from this solution on boiling. It is insoluble in alcohol and ether. When heated, it yields titanic acid, hydrogen chloride, and water. F. S. K.

Titanium Trioxide. By A. Piccini (*Ber.*, **21**, 1391—1393), and by A. Classen (*Ber.*, **21**, 1519).—A question of priority.

Thio-derivatives of Antimonic Acid. By W. Feit and K. Kubierschky (*Ber.*, **21**, 1660—1662).—Attempts were made to prepare thio-derivatives of antimonic acid:—(1.) By adding antimony pentasulphide to strong aqueous soda until a portion remained unacted on; on adding alcohol to the solution, sodium tetrathioantimoniate separated in well-formed tetrahedra, no other compound being formed. (2.) By adding antimony sulphotrichloride to aqueous soda. (3.) By the action of lead oxide on sodium tetrathioantimoniate. (4.) By the action of antimony oxytrichloride on sodium sulphide. (5.) By boiling a solution of sodium metantimoniate with sodium sulphide. In (2) and (4) the tetrathioantimoniate was formed. The author concludes that thio-derivatives of antimonic acid containing less than 4 atoms of sulphur cannot exist. N. H. M.

The Anomalous Density of Liquid Bismuth. By C. Lüdeking (*Ann. Phys. Chem.* [2], **34**, 21).—The author finds that, like water, liquid bismuth attains a maximum density just before becoming solid. The expansion from the point of maximum density the author finds from the mean of two determinations to be 0·0000864, and the expansion at the moment of solidification to be about 3 per cent. of the volume, or about a third of that of water. Vincentini (*Atti R. Acc. Sc. Torino*, **22**, 23) gives a value three times as great for the instantaneous expansion, and was unable to find any expansion before solidification. He however used a dilatometer much less delicate than that employed by the present author, and the latter ascribes the high value obtained by Vincentini for the expansion on solidifying, to the presence of air in his dilatometer, as he considers that this could not be avoided in the method used by him. G. W. T.

Sodium Platosammonium Sulphite. By W. Haberland and G. Hanekop (*Annalen*, **245**, 235—238).—When a solution of chloroplatinic acid is saturated with sulphur dioxide and the liquid neutralised with sodium carbonate, a white precipitate is thrown down which is soluble in hydrochloric acid. On the addition of ammonia in excess to the acid solution, rhombic crystals of sodium platosammonium sulphite, $2NH_3,Pt(SO_3Na)_2 + 5\frac{1}{2}H_2O$, are slowly deposited. This salt is analogous to the ammonium salt described by Peyrone (*Gmelin-Kraut's Handbuch*, **3**, 1099). W. C. W.

Rhodium Sesquichloride. By E. Leidié (*Compt. rend.*, **106**, 1076—1079).—When rhodium is heated in chlorine to incipient redness, the lowest temperature at which the action proceeds readily, the proportion of chlorine in the product never approaches that existing in the sesquichloride, and is less the higher the temperature. With a rapid current of chlorine at bright redness, a deposit with the colour of peach-blossom forms on the cold part of the tube, but it is a product of dissociation, and its composition is variable. At 350° or 440°, chlorine attacks rhodium very slowly.

Fellemberg's method of heating rhodium sulphide in chlorine yields no definite results, and a temperature sufficient to secure the complete expulsion of the sulphur decomposes the chloride. The action of sulphuric acid on the rhodiochlorides yields rhodium sulphate, and not, as Claus states, rhodium chloride.

Hydrated rhodium sesquichloride, or hydrated ammonium rhodiochloride, when heated in pure and dry nitrogen at 440°, yields only rhodium sesquioxide, hydrogen chloride, or hydrogen chloride and ammonium chloride being evolved.

The best method of obtaining anhydrous rhodium sesquichloride is to heat the alloy, $RhSn_3$, described by Debray (Abstr., 1887, 779), in a current of chlorine at 440°, a temperature at which the chloride does not dissociate. Stannous chloride volatilises. The product is allowed to cool completely in a current of chlorine, and this gas is then displaced by carbonic anhydride. Rhodium sesquichloride thus obtained is a brick-red powder, which retains the crystalline form of

the alloy. The results of the analyses agree exactly with the formula Rh_2Cl_6.

The author has prepared and examined a number of rhodiochlorides.

Ammonium rhodiochloride, $Rh_2Cl_6,6NH_4Cl + 3H_2O$, has the crystalline form described by Keferstein. The author was unable to obtain the chloride, $Rh_2Cl_6,4NH_4Cl + 2H_2O$, described by both Vanquelin and Claus, who, however, differ considerably in their description. The sodium salt has the composition $Rh_2Cl_6,6NaCl + 18H_2O$. The potassium salt, $Rh_2Cl_6,4KCl$, crystallises in the rhombic system as stated by Berzelius, but it contains no water. The hydrated chloride, $Rh_2Cl_6,6KCl + 6H_2O$, described by Claus, could not be obtained. C. H. B.

Mineralogical Chemistry.

Phosphorescent Blende. By A. Verneuil (*Compt. rend.*, **106**, 1104—1107).—Natural hexagonal crystals of blende have comparatively feeble phosphorescent properties. If natural zinc sulphide is distilled in a slow current of hydrogen some hydrogen sulphide is evolved, and small hexagonal crystals of zinc sulphide are obtained, which show a brilliant green phosphorescence. The same result is obtained by distilling artificial zinc sulphide, but non-phosphorescent hexagonal crystals are also obtained. The crystals which form in the front part of the tube always show the most brilliant phosphorescence.

The phosphorescence is not due to impurities in the hydrogen, nor yet to impurities derived from the tube, since the same results are obtained if the zinc sulphide is heated in a platinum boat. Variations of temperature between an orange-red heat and a full white heat are without influence, and variations in the velocity of the current of hydrogen likewise have no material effect.

If zinc sulphide is heated in a platinum dish in a stationary atmosphere of hydrogen, hydrogen sulphide is evolved and some metallic zinc is obtained, and the product is not phosphorescent. With a moderately rapid current of gas, the phosphorescence appears, and with a rapid current the same results are obtained as when the zinc sulphide is in contact with the sides of the tube.

C. H. B.

Analyses of Franklinite. By G. C. Stone (*Zeit. Kryst. Min.*, **14**, 291—292, from *School of Mines Quarterly*, **8**, 148—152).—The author has analysed a series of specimens picked from ore from the two mines of the New Jersey Zinc and Iron Company, in Sussex Co., New Jersey. The ore from the Sterling Hill Mine, near Ogdensburg, is mostly franklinite with calcite and some zincite, whilst that

from the Taylor mine at Franklin Furnace is mainly franklinite, willemite, and zincite. Analysis gave the following results:—

	I*a*.	I*b*.	II*a*.	II*b*.	II*c*.	II*d*.
Fe_2O_3.....	0·44	0·36	67·38	66·34	60·52	56·57
Mn_2O_3 ...	—	—	—	—	6·79	10·52
MnO	6·50	5·54	16·38	12·31	12·81	16·37
ZnO	93·28	94·30	16·28	20·26	19·44	15·91
Totals	100·22	100·20	100·04	98·91	99·56	99·37
Sp. gr.....	5·53	5·51	5·21	5·07	5·19	5·14

I. Zincite, *a*, dark-red grains; *b*, dark-red round lamellar mass. II. Franklinite. Of this mineral, the author finds two distinct varieties, both corresponding with the general formula RO,R_2O_3. One variety (II*a* and *b* from Sterling Hill) has a dull lustre, and dissolves with difficulty in hydrochloric acid without any evolution of chlorine, whilst the other (II*c* and *d* from Taylor mine) has more lustre, and dissolves very readily in hydrochloric acid with evolution of chlorine. The following are analyses of willemite from the Taylor mine: *a*, nearly white, dull; *b*, pale-green, vitreous; *c*, orange, dull; *d*, greenish-yellow to bright-yellow, vitreous; *e*, dark-red, dull.

	III*a*.	III*b*.	III*c*.	III*d*.	III*e*.
SiO_2	27·20	26·92	28·30	27·48	27·14
ZnO.....	65·82	65·04	66·68	63·88	64·38
MnO.....	6·97	7·78	4·92	8·33	6·30
FeO.....	0·23	0·51	0·31	0·49	1·24
Totals	100·22	100·25	100·21	100·18	99·06
Sp. gr.....	4·19	4·19	4·18	4·16	4·13

Tephroite gave the following results:—

SiO_2.	MnO.	ZnO.	FeO.	MgO.	Total.
30·63	49·80	5·74	3·33	10·16	99·66

B. H. B.

Pseudomorph of Manganite: Artificial Formation of Pyrolusite. By A. Gorgeu (*Compt. rend.*, **106**, 1101—1104).—When manganite, Mn_2O_3,H_2O, is heated for about seven hours at 270—310°, in presence of air, it is completely converted into the peroxide. With the crystallised mineral, the change requires a longer time. The crystals retain their metallic lustre, but the colour changes from violet to iron-grey. The hardness 3·0 is sensibly equal to that of pyrolusite, and a similar agreement is observed between the specific gravities. Direct measurements show that the crystals retain their original form. It follows that under the conditions described,

polianite is obtained as a pseudomorph after manganite. In other words, manganese peroxide has been prepared artificially with a form and properties common in the natural mineral.

Direct experiment shows that the conversion of manganite into the peroxide by heating in contact with air is practicable on a large scale, with a corresponding increase in the oxidising value. The mineral must be heated gradually, so that the water is expelled slowly, otherwise the absorption of oxygen is comparatively small. Braunite, hausmannite, and psilomelane do not absorb oxygen in notable quantity between 200° and 425°. Manganous carbonate under these conditions oxidises slowly and incompletely.

C. H. B.

Lansfordite, a New Mineral. By F. A. Genth (*Zeit. Kryst. Min.*, **14**, 255—256).—This interesting mineral was found, in small stalactites, in a fissure in the roof of a gallery in the Lansford anthracite mine, near Tamaqua, in Schuylkill Co., Pennsylvania. The stalactites are white and transparent, and exhibit crystalline structure. Their hardness is 2·5, and their sp. gr. 1·692. Analysis gave results corresponding with the formula $3MgCO_3,Mg(OH)_2 + 21H_2O$.

B. H. B.

Occurrence of Thenardite in Russia. By W. Markownikoff (*J. Russ. Chem. Soc.*, 1887, 245—252).—It is known that in the bitter lakes of South Eastern Russia (on the north of the Caspian Sea, and east of the mouth of the Volga) sodium sulphate occurs in solution, and in 1884 the author showed that the principal constituent is astrachanite; he has now ascertained that anhydrous sodium sulphate, thenardite, also occurs in a nearly pure state, being formed in the lakes under the influence of the high summer temperature. The author finds that the quantity of magnesium (and of astrachanite) in the salts (A) is greater near the delta of the Volga, and less (B) as we proceed in an easterly direction; this is shown by the following analyses:—

	Na_2SO_4.	$MgSO_4$.	$CaSO_4$.	NaCl.	Insoluble residue.	H_2O.	Total.
(A.)	74·95	3·97	1·44	2·62	0·89	17·00	99·88
(B.)	96·31	0·69	0·22	2·31	0·29	0·10	99·92

Thenardite is also found in the Caucasus; this, however, is not of recent date, like that from the Astrachan lakes, but belongs to a former geological epoch. A sample was analysed by the author:—

Na_2SO_4.	$CaSO_4$.	H_2O.	Total.
99·69	0·28	0·15	100·12 per cent.

Thenardite is also found in the Kirgisian mountain, Tchaptchali, between rock salt layers, in the permian formation. The author thinks that the occurrence of thenardite in the said lakes is due to the contact of water with these layers, as the mother-liquor from which thernadite is deposted, according to his analyses, does not

contain a sufficient quantity of magnesium salt to warrant the assumption that it has been formed by double decomposition from magnesium sulphate and sodium chloride. B. B.

Dihydrothenardite, a New Mineral. By W. MARKOWNIKOFF (*J. Russ. Chem. Soc.*, 1887, 252—254).—This salt, $Na_2SO_4 + 2H_2O$, has been found in the Lake Gori, of the Tiflis Government. It is found on the shore as a layer, 1 cm. in thickness, and is even in outward appearance different from astrachanite, thenardite, and mirabilite. Its structure appears to be crystalline, the fracture is colourless, but the surface is opaque from slight efflorescence, which, however, does not go on very quickly. Its analysis gave the following result:—

Water at 250°.	Na_2SO_4.	NaCl.	Total.
16·15	81·18	0·98	98·32

The difference from 100° is due to traces of clay, gypsum, and magnesium sulphate. The crystalline form was determined by G. N. Wyrouboff, of Paris, who in vain tried to obtain this new hydrate artificially. In the purest sample, 19·6 per cent. of water was found (instead of 20·2 as required by theory). The crystals are monoclinic; $a : b : c = 0·4651 : 1 : 0·7194$; $\gamma = 101° 5'$. It is probable that salt A quoted in the preceding paper consists chiefly of dihydrothenardite. B. B.

Pharmacolite. By H. DUFET (*Compt. rend.*, **106**, 1238—1240).—A vessel containing a solution of disodium arsenate was placed inside a vessel containing a solution of calcium nitrate, water was added, and the two salts were allowed to mix by diffusion. Under these conditions well-defined, monoclinic prisms of pharmacolite are obtained, identical in form with the crystals described by Haidinger and Schrauf. They have the composition $CaHAsO_4 + 2H_2O$, not $2CaHAsO_4 + 5H_2O$, as stated by Klaproth and by Rammelsberg, and they are isomorphous with brushite, $CaHPO_4 + 2H_2O$. Details of the measurements of the crystals are given. C. H. B.

A Crystallised Slag. By F. FOUQUÉ (*Zeit. Kryst. Min.*, **14**, 283—284, from *Bull. soc. fran. min.*, **9**, 287).—A slag from the St. Nazaire Works contained a crystallised substance, whose form was cubic, whilst its optical properties were partly those of a positive uniaxial crystal and partly anomalous. Its hardness was less than 5, and its sp. gr. 2·91. Analysis gave the following results:—

SiO_2.	Al_2O_3.	CaO.	MgO.	Fe_2O_3.	Total.
37·60	12·26	40·11	9·33	trace	99·30

This composition is nearly that of melilite, which, however, contains less silica, and has a negative double refraction. It also invariably contains ferric oxide and alkalis. B. H. B.

Spodumene from Brazil. By P. JANNASCH (*Jahrb. f. Min.*, 1888, i, Mem., 196—201).—The author has made a very careful analysis of transparent, yellowish-green spodumene from Brazil, the results being as follows:—

SiO_2.	Al_2O_3.	FeO.	MnO.	CaO.	Li_2O.	Na_2O.	H_2O.	Total.
64·32	27·79	0·67	trace	0·17	7·45	0·55	0·12	101·07

The sp. gr. is 3·174. These analytical results are in accord with Doelter's formula for spodumene, $Li(Na)_2Al_2Si_4O_{12}$. Compared with recent analyses of spodumene, this analysis shows the highest percentage of lithia and the lowest of soda. B. H. B.

Products of the Alteration of Rhodonite. By A. H. CHESTER (*Jahrb. f. Min.*, 1888, i, Mem., 187—190).—A specimen of manganese ore, resembling v. Kobell's klipsteinite, was found by the author to be an altered rhodonite. The results of the analysis of the portion soluble in hydrochloric acid (I*b*) and of the insoluble portion (I*a*) are given. The substance is undoubtedly a mixture of two minerals, the soluble one being formed by the combination of the other with oxygen and water.

	I*a*.	I*b*.	II*a*.	II*b*.	III.
SiO_2	26·22	—	43·31	0·23	48·75
MnO	15·54	—	15·49	—	30·66
FeO	6·44	—	8·44	—	10·85
CaO	0·75	0·90	2·20	1·82	6·21
MgO	—	4·24	11·74	4·89	0·91
Mn_2O_3	—	30·07	—	4·74	—
$Fe_2O_3 + Al_2O_3$	—	7·95	—	4·65	—
CO_2	—	—	—	—	1·70
H_2O	—	7·94	—	2·25	0·80
Total	—	100·05	—	99·76	99·88

The analysis shows that the insoluble portion consists of rhodonite in which FeO is substituted for a portion of the MnO, and that the soluble portion is wad. The mineral is therefore merely a mixture of these two.

A mineral from Cumberland, Rhode Island, known as marceline, was analysed in the same way. The insoluble portion (II*a*) is rhodonite, in which MnO is largely replaced by FeO and MgO. The soluble portion (II*b*) is wad containing a large portion of lime and magnesia.

A specimen of so-called photicite (Analysis III) from Cumberland, Rhode Island, is obviously nothing more than a slightly altered rhodonite. None of these three minerals therefore deserves a special name; they should all be classed as more or less altered rhodonite.

B. H. B.

Clay Slate and Sericite Slate. By A. v. GRODDECK (*Jahrb. f. Min.*, 1888, i, Ref., 415—417; from *Jahrb. k. preuss. geol. Landes-anst. für* 1885, 1—52).—The author gives 17 analyses of clay slates from the

Upper Harz. The microscopical examination showed that the Culm clay slate and the Devonian clay slate contained quartz, a sericite-mica, and a chloritic mineral as principal constituents. Carbonaceous matter, carbonates, and needles of rutile were also observed. Black vein-clay (the soft clay lying between the ore and the sides of the vein) exhibits as a rule the same mineralogical constitution. The coloured vein-clays consist only of quartz and sericite, needles of rutile, and a quantity of iron ore (goethite?). The alteration of clay state to vein-clay seems to be due to a decrease of the chloritic constituents, which in the former average 16·54 per cent., and fall to 4·37 per cent. in the black vein-clay and to zero in the coloured variety. B. H. B.

Analysis of Roncegno Water. By M. Gläser and W. Kalmann (*Ber.*, **21**, 1637—1639).—The water from Roncegno (South Tyrol) is yellow from the presence of ferric sulphate. The following numbers which show the weight in grams per litre of water at 18° of the several constituents form the mean of two series of analyses:—

SiO_2.	SO_3.	As_2O_5.	P_2O_5.	Cl.	CuO.	FeO.
0·1283	4·4675	0·1621	0·0123	0·0027	0·0037	0·0303

Fe_2O_3.	MnO.	CoO.	NiO.	Al_2O_3.	CaO.
1·2495	0·1087	0·0114	0·0381	0·4343	0·7821

MgO.	K_2O.	Na_2O.	Organic matter.
0·1210	0·0163	0·1270	0·2246

Total solids = 7·9396, sp. gr. = 1·00748 at 18°. N. H. M.

Organic Chemistry.

Volatility of Poly-oxygen Carbon-compounds. By L. Henry (*Compt. rend.*, **106**, 1089—1092).—A comparison of the boiling points of hydrocarbons, ketones, and polyketones shows that a double substitution of O for H_2 causes a much smaller proportional increase in the boiling point than a single substitution in the same part of the hydrocarbon-chain. The second substitution of *O* for H_2 in a hydrocarbon in the same portion of the chain as the first substitution causes a very much smaller rise in the boiling point. This is well shown by the following compounds:—

	B. p.		B. p.
Normal butane.....	+ 1°	Normal pentane.....	39°
Methyl ethyl ketone ..	78	Methyl propyl ketone .	101
Diacetyl	88	Acetylacetone	137

Hexane.....	68°
Methyl butyl ketone ..	127
Acetonylacetone	187

In the butane series, the first substitution increases the molecular

weight 24·13 per cent., and the absolute boiling point 28·10 per cent., whilst the second substitution raises the molecular weight 19·44 per cent., but the boiling point only 2·84 per cent.

Methyl ethyl ketone and pentane have the same molecular weight, but the substitution of O for H_2 in the latter raises the boiling point 64°, but in the former only 10°.

It follows that the simultaneous presence of several oxygen-atoms in the same part of the molecule tends to reduce the boiling point.

C. H. B.

Volatility of Oxygenised Carbon-compounds. By L. Henry (*Compt. rend.*, **106**, 1165—1167).—The influence of oxygen in increasing the volatility of carbon-compounds is most strongly marked when two atoms of oxygen are directly united with one atom of carbon as in carbonic anhydride. The conversion of methane into carbonic anhydride increases the molecular weight by 175 per cent., but the absolute boiling point by only 77·06 per cent. When the two oxygen-atoms are united separately to different carbon-atoms, as in the group —CO—CO—; the latter, however, being in direct union with one another, the effect, although smaller, is still strongly marked. The conversion of normal butane into diacetyl raises the molecular weight 48·27 per cent. and the absolute boiling point 31·75 per cent. The volatilising effect is still evident when the two carbon-atoms are separated by a third carbon-atom as in the chain —CO—CH_2—CO—, but it disappears when two atoms intervene. The conversion of normal pentane into acetylacetone raises the molecular weight 38·88 per cent. and the absolute boiling point 32·25 per cent. Hexane and methyl propyl ketone have the same molecular weight, but the conversion of the former into acetylacetone raises the absolute boiling point 9·62 per cent., whilst the corresponding substitution in the hydrocarbon causes an increase of 17·30 per cent.

The volatilising influence of nitrogen is greater than that of oxygen, but it is affected to a much greater extent by separation of the carbon-atoms with which the nitrogen-atoms are united.

C. H. B.

Action of Feeble Bases on Nitroethane. By N. Sokoloff (*J. Russ. Chem. Soc.*, 1887, 384—387).—In a former paper, the author noticed that the action of ammonia in alcoholic solution on nitroethane gave rise to a compound, C_6H_9NO, and now he finds that a large quantity of ethaldehyde is formed at the same time. B. B.

Some Properties and Transformations of Dimethylallene (Reboul's Valerylene). By A. Albitzky (*J. Russ. Chem. Soc.*, 1887, 364—368).—Bromide of trimethylethylene (b. p. 115—125°) was prepared by the action of bromine on dimethyl ethyl carbinol, and this, by treatment with alcoholic potash, gave trimethylethylene. The bromide of trimethylethylene was heated in sealed tubes for 12 hours at 165° with thrice its bulk of alcoholic potash of 50 per cent., and some solid potash. The contents of the tubes, after being distilled on the water-bath, were decomposed by water, and the upper oily layer was washed with water, and dried over sodium sulphate. On repeated distillation over

caustic potash and metallic sodium, the hydrocarbon was obtained as a liquid boiling at 40·5—41·5°. The yield is one-third of the theoretical. It is *dimethylallene*, C_5H_8. Its density (water at 4° = 1) at 0° = 0·7525, at 17° = 0·6970, at 20° = 0·6940. Coefficient of dilatation between 0° and 20° = 0·0014. Optical constants: $\frac{A-1}{d_4^{17}} = 0{\cdot}57477$, $p\frac{A-1}{d_4^{17}} = 39{\cdot}08$. Calculated, 34·62; difference, 4·46. Lorenz's formula, $\frac{A^2-1}{(A^2+2)d} = 0{\cdot}34829$, $p\frac{A^2-1}{(A^2+2)d} = 23{\cdot}68$. Calculated, 20·31; difference, 3·37. Two double linkings require a difference of 3·18. Oxidation with chromic mixture at the ordinary temperature after 25 days yielded acetone and acetic acid. The structure of dimethylallene is therefore $CMe_2{:}C{:}CH_2$. Sulphuric acid, sp. gr. 1·64, at the ordinary temperature yielded methyl isopropyl ketone and probably also divalerylene hydrate or trivalerylene. Dilute acid gives no better result. B. B.

Isomeric Change of Hydrocarbons of the Acetylene Series by Heating them with Alcoholic Potash. By A. Favorsky (*J. Russ. Chem. Soc.*, 1887, 414—427).—Methyl ethyl ketone was acted on with phosphorus pentachloride, and the chloride, $CH_2Me{\cdot}CMeCl_2$, obtained was heated in a sealed tube with alcoholic potash at 170°, a temperature which, according to Kutscheroff, is best adapted for the formation of ethylacetylene. The hydrocarbon obtained on distilling the contents of the tubes did not reduce ammoniacal silver or cuprous chloride solution, and when heated with sulphuric acid, crystals of hexamethylbenzene (m. p. 163·5°) were obtained. This shows that instead of ethylacetylene, $CH_3{\cdot}CH_2{\cdot}C{:}CH$, as expected, dimethylacetylene, $CH_3{\cdot}C{:}C{\cdot}CH_3$, is formed; ethylacetylene is obtained, however, if dry potassium hydroxide is substituted for its alcoholic solution. When pure ethylacetylene is heated with pure ethyl alcohol, no isomeric change takes place, showing that alcohol alone has not caused the change. If, however, the hydrocarbon is heated for 16 hours at 170° with alcoholic potash, it will be found to be converted into dimethylacetylene, as is shown by the formation of hexamethylbenzene (m. p. 163·5°) when it is treated with sulphuric acid (5 to 1 water); ethylacetylene under these circumstances does not give hexamethylbenzene. These facts show that the isomeric change has taken place under the influence of alcoholic potash.

In order to subject propylacetylene to the action of alcoholic potash, the product of the action of phosphorus pentachloride on methyl propyl ketone, $CH_3{\cdot}CCl_2{\cdot}CH_2{\cdot}CH_2{\cdot}CH_3$, was purified and heated with alcoholic potash at 120°. The propylacetylene, $CH_3{\cdot}CH_2{\cdot}CH_2{\cdot}C{:}CH$, obtained, was purified by converting it into the cuprous compound, decomposing the latter with hydrochloric acid and distilling. The purified hydrocarbon was then heated with alcoholic potash at 170°, when it was found to be converted into the isomeric methylethylacetylene, $CH_3{\cdot}CH_2{\cdot}C{:}C{\cdot}CH_3$, which gives no precipitate with ammoniacal cuprous or silver solutions and boils at 55·5—56°. The same product

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is obtained by the direct action of alcoholic potash at 150° on the chloride. On oxidation with chromic mixture, methylacetylene yields acetic and propionic acids; with sulphuric acid (1 water to 5 of acid), methyl propyl ketone was obtained, so that the above formula is confirmed. The isomeric change begins to take place at as low a temperature as 30—40°.

When isopropylacetylene, as a hydrocarbon of the acetylene series containing a secondary radicle, was heated with alcoholic potash at 150°, dimethylallene, $CMe_2{:}C{:}CH_2$, was obtained; this was proved to be identical with that obtained from trimethylethylene. According to the author's theoretical views, a hydrocarbon of the acetylene series containing a tertiary group should not be altered by alcoholic potash. In order to prove this, hexoylene, $CMe_3{\cdot}C{:}CH$, was prepared from the dichloride from pinacoline by heating it with alcoholic potash at 140°; the hydrocarbon boils at 39°, and gives both silver and cuprous compounds like a true acetylene. When heated with alcoholic potash, first for 8 hours at 170°, and then again for 16 hours at 200°, no alteration takes place. The isomeric change of monosubstituted acetylenes by the action of alcoholic potash is found to depend not only on their structure, but also on their molecular weight; caprylidene, for example, is obtained even at 150° (Béhal), whereas ethylacetylene and propylacetylene are easily converted into isomerides under the same conditions. B. B.

Pyrrolylene Tetrabromides. By G. Ciamician and G. Magnanini (*Ber.*, **21**, 1430—1434).—The authors have repeated Ciamician and Magnaghi's work (Abstr., 1886, 521), with the result that Grimaux and Cloez' conjecture (Abstr., 1887, 789) as to the source of the pyrrolylene (erythrene) tetrabromide melting at 38—39° is shown to be untenable. Pyrrolylene, obtained from erythrol, was brominated in the cold, and the product freed from excess of bromine by a current of air was extracted with light petroleum, in which Henninger's tetrabromide (m. p. 118—119°) is practically insoluble. On spontaneous evaporation of the solution, an oil was obtained which solidified in a freezing mixture, and, after removal of a small quantity of adhering oil, crystallised from light petroleum in colourless tables melting at 38—39°: hence the tetrabromide of lower melting point is a direct product of the bromination of pyrrolylene. The proportion in which the isomeric tetrabromides are formed is not always the same, and the quantity of the tetrabromide melting at 38—39° obtained in these experiments was less than in those of Ciamician and Magnaghi.

Pyrrolylene tetrabromide (m. p. 118—119°) crystallises in monoclinic forms: $a : b : c = 2{\cdot}63485 : 1 : 2{\cdot}333815$; $\beta = 80° 55'$. It is not converted into its isomeride by prolonged heating with excess of bromine at 100°, and when distilled at 180—181° under 60 mm. pressure, yields a distillate from which traces only of the tetrabromide of lower melting point could be extracted by light petroleum.

W. P. W.

Action of Zinc Cyanide on Metallic Chlorides. By R. Varet (*Compt. rend.*, **106**, 1080—1083).—A concentrated aqueous

solution of mercuric chloride is heated at 50—60° and mixed with pure zinc cyanide in quantity equal to about half the weight of the mercuric chloride. The liquid is then saturated with a further quantity of mercuric chloride, mixed with zinc cyanide in excess, filtered, and allowed to evaporate over sulphuric acid. The compound, $ZnCy_2,HgCy_2,HgCl_2 + 6H_2O$, separates in large, transparent, efflorescent, prismatic crystals, which become anhydrous at 100°; they are very soluble in water. When treated with gaseous ammonia, the compound becomes anhydrous and combines with 3 mols. of ammonia. When treated with aqueous ammonia, the crystals become white and dissolve with some development of heat; and when the solution cools, it deposits small, crystalline nodules of the compound $ZnCy_2,HgCy_2,HgCl_2,6NH_3$, which loses ammonia when exposed to air, and is decomposed by water. It is slightly soluble in cold aqueous or alcoholic ammonia, and dissolves readily on heating.

When a solution of the first compound is mixed with potassium iodide, zinc cyanide is first precipitated, and afterwards mercuric iodide. By rapid manipulation, the zinc cyanide can be removed before the mercuric iodide separates. The presence of potassium iodide causes a reaction the reverse of that by which the compound was formed. Potassium iodide combines with mercuric cyanide, but not with zinc cyanide. If metallic copper is immersed in the ammoniacal solution, mercury is deposited, and the solution becomes blue. When concentrated, the liquid yields blue crystals which contain zinc, copper, and mercury. Copper does not displace mercury from mercuric cyanide, and from these results it follows that the compound has the constitution represented by the formula given, and cannot be regarded as a compound of mercuric cyanide with zinc chloride.

When zinc cyanide is added to a concentrated solution of cupric chloride, brown cupric cyanide is precipitated, heat is developed, and the cupric cyanide becomes cuprous cyanide with evolution of cyanogen. This reaction affords a convenient method for the preparation of pure cyanogen, and zinc cyanide has a great advantage over potassium cyanide in that it is not so liable to be contaminated with carbonate. When dry zinc cyanide and cupric chloride in equivalent proportions are heated together in an oil-bath, the evolution of cyanogen begins at about 150° and proceeds regularly at 160—170°. Zinc cyanide alone decomposes at about 300°.

Chlorides of the alkalis and alkaline earths, nickel, manganese, cobalt, iron, gold, platinum, &c., form no compounds with zinc cyanide, but yield a series of double salts with ammonio-zinc cyanide.

Zinc cyanide forms no double salts with metallic chlorides only.

C. H. B.

Action of Sodium on Homologues of Ethyl Cyanide. By E. v. Meyer (*J. pr. Chem.* [2], **37**, 396—407).—The author has previously studied the formation and reactions of cyanethine and cyanmethine (Abstr., 1881, 54; 1883, 352, 653). *Cyanopropine*, $C_{12}H_{21}N_3$, is obtained by adding 77 grams of propyl cyanide to 8 grams of sodium in a retort closed with mercury; a violent action ensues, and propane is evolved. When all the sodium has disappeared, the

excess (23 grams) of cyanide is distilled off, and the residue washed with water, dried, and recrystallised from hot ether, which deposits it on cooling in pointed prisms. It melts at 115°, and is soluble in 1572 parts of water at 23°, forming a distinctly alkaline solution. Its platinochloride crystallises in slender, yellow-red prisms melting at 97°. By heating cyanopropine with concentrated hydrochloric acid at 180° for some hours, and then neutralising with sodium hydroxide, a hydroxy-base, $C_{12}H_{19}N_2{\cdot}OH$, is obtained in slender needles; this melts at 97·5°, and is soluble in 1497 parts of water at 23°; it is easily soluble in alkalis, and gives an amorphous silver salt, $C_{12}H_{19}AgN_2O$, when a mixture of its solution with silver nitrate is neutralised with ammonia.

Bromocyanopropine crystallises as its hydrobromide, when an acid solution of cyanopropine is evaporated with bromine, and from this it may be obtained on the addition of ammonia. It forms needles melting at 80°. During the action of the bromine, an oil is obtained which behaves with ammonia like the similar product from cyanethine and bromine (Abstr., 1883, 353), giving a sparingly soluble amide of symmetrical diethylsuccinic acid.

Isopropyl cyanide acts very violently on sodium, yielding a slimy, brick-red mass, which, after the excess of the cyanide has been distilled off, is treated with water, whereupon it evolves ammonia, leaving a disagreeably smelling oil undissolved. This oil partially crystallises after a time; recrystallisation from benzene gives slender needles of the composition $C_{19}H_{34}N_6$, melting about 241°; the platinochloride forms thin, yellow prisms sparingly soluble in water and containing $2\frac{1}{2}$ mols. H_2O, of which $1\frac{1}{2}$ mols. are retained at 170°. The portion of the oil that does not crystallise gives off isopropyl cyanide and water at 87°; treatment of the residue with concentrated hydrochloric acid at 60° gives an oil, which may be extracted by ether, and an acid solution. The dried oil gives two fractions which are split up into isobutyric acid and ammonia by acid and alkalis, and a third from which di-isobutyramide (Abstr., 1882, 950) is obtained by the action of concentrated hydrochloric acid. The acid solution from which the oil was extracted contained ammonium isobutyrate, and gives a mixture of oils when neutralised with potassium hydroxide; when this mixture is treated with concentrated hydrochloric acid at 190°, ammonium chloride is formed, and by distilling the acid liquid with excess of alkali, it yields a base. This base, $C_{12}H_{22}N_2$, forms silky needles melting at 136—137°, subliming in prisms at 100°, sparingly soluble in water to a distinctly alkaline solution, freely soluble in alcohol and ether. Its platinochloride, $(C_{12}H_{22}N_2)_2,H_2PtCl_6$, forms thin, yellow prisms; and its silver nitrate compound, $(C_{12}H_{22}N_2)_2,AgNO_3$, a voluminous, white precipitate. By oxidation with permanganic acid, the base is converted into di-isobutyramide, ammonia, carbonic anhydride, isobutyric acid, and isopropyl cyanide. A. G. B.

Action of Sodium on Isobutyl Cyanide and Isoamyl Cyanide. By J. Troeger (*J. pr. Chem.* [2], **37**, 407—411).—*Cyanobutine*, $C_{18}H_{27}N_3$, is obtained when 40 grams of isobutyl cyanide and 4 grams of sodium are gently heated together in a retort until action begins. A gas, probably trimethylmethane, is evolved; the residue is after-

wards treated with water. The undissolved resin is dissolved in ether, and hydrogen chloride passed through the solution. The hydrochloride thus formed is decomposed by alcoholic ammonia, and the resulting oil extracted by ether; the ether is evaporated, and leaves an oil which gradually crystallises in stellate groups. A *hydroxy-base*, $C_{15}H_{25}N_2{\cdot}OH$, is obtained by heating cyanobutine with hydrochloric acid, or by passing nitrous anhydride through a solution of it in glacial acetic acid, and adding ammonia, when it is precipitated as an oil, which crystallises on cooling, and melts at 88—89°.

Methylcyanobutine hydriodide, $C_{15}H_{26}MeN_3{\cdot}HI$, is obtained when cyanobutine is heated with methyl iodide at 160°.

Cyanamyline, $C_{18}H_{33}N_3$, is obtained by heating 40 grams of isoamyl cyanide with 3·5 grams of sodium; after distilling off excess of the cyanide and washing with water, the residue is dissolved in alcohol; on dilution with water the base is thrown down as an oil, crystallising gradually. The crystals are purified by distillation. Cyanamyline is a strong base, melting at 53°.

A *hydroxy-base*, $C_{18}H_{31}N_2{\cdot}OH$, is obtained when nitrous anhydride is added to the solution of cyanamyline in glacial acetic acid. It forms long, white needles, and has only feebly basic properties.

A. G. B.

Formation of Cyanethine from Ethyl Cyanide. By E. v. Meyer (*J. pr. Chem.* [2], **37**, 411—413).—In this preliminary paper the author reverts to his previous work (Abstr., 1881, 54). Cyanethine is not obtained by the action of sodium on an ethereal solution of ethyl cyanide, but a white powder is formed, which partially dissolves in water, and from the solution an oil separates, which eventually crystallises. This new substance is a polymeride of ethyl cyanide; it melts at 47—48°, and boils between 257° and 258° unchanged; it has hardly any basic properties, and is converted by acids into ammonia and a limpid oil containing nitrogen, which has a strong reducing action on silver nitrate. Sodium acts energetically on this polymeride when heated with it, forming ethyl cyanide, which is partially converted into cyanethine. The author and others are trying the action of sodium on ethereal solutions of other alkyl cyanides.

A. G. B.

Tertiary Ethyl Amyl Ether. By J. Kondakoff (*J. Russ. Chem. Soc.*, 1887, 300—301).—Tertiary amyl ethyl ether is obtained as a bye-product when trimethylethylene is prepared from tertiary amyl iodide by Boutleroff's method. It remains dissolved in the alcoholic potash, and may be separated by the addition of water; the yield is very small, 20 grams only having been obtained from 900 grams of iodide. It boils at 102°, and is a light, mobile liquid of sp. gr. 0·7785 at 0°, and 0·751 at 18°. As it yields ethyl iodide and tertiary amyl iodide when treated with hydriodic acid, its constitution is $C_5H_{11}{\cdot}OEt$. The ether has been already described by Réboul (*Compt. rend.*, **64**, 1243), but as in Beilstein's *Handbuch*, the name of ethyl amyl ether is given to the ether of methyl isopropyl carbinol, the author has tried to prepare his ether synthetically from tertiary amyl iodide

and sodium ethoxide; the yield, however, was very small. The same compound was prepared from the iodide of methyl isopropyl carbinol and sodium ethoxide, but in both reactions large quantities of trimethylethylene were obtained. B. B.

Action of Halogen Hydrides on Oxymethylene. By W. Tishtchenko (*J. Russ. Chem. Soc.*, 1887, 464—478).—In 1883, the author found that on heating oxymethylene with aqueous halogen acids, formic acid was formed, together with halogen-derivatives of methane. In order to study this reaction, in which both water and the halogen hydride seem to take a part, the author has heated oxymethylene with water for several days in sealed tubes at 115°. The product of the reaction contained no formic acid, and the reaction was therefore not analogous to that which takes place with halogen acids, for only a little methyl alcohol was formed, as it would seem owing to the presence of alkalis from the glass. When oxymethylene was heated with very weak halogen acids, it remained nearly unaltered even for weeks, and only very small quantities of methyl chloride and formic acid were found among the products of reaction. In order to study the action of dry halogen hydrides on oxymethylene, an apparatus was employed, which could be weighed. After bringing in dry oxymethylene, dry hydrogen iodide was passed over it, when a reaction accompanied by the development of heat began, so that after 30 minutes the oxymethylene disappeared; the hydride, however, does not cease to be absorbed until after 12 to 16 hours. The product of the reaction consists of two layers, of which the upper is aqueous hydriodic acid, and the lower a liquid, boiling at 219°, and having the composition $C_2H_4I_2O$, that is, the di-iodated methyl ether, the formation of which the author represents by two phases: (*a*) $CH_2O + HI = CH_2I{\cdot}OH$, and (*b*) $2CH_2I{\cdot}OH = CH_2I{\cdot}O{\cdot}CH_2I + H_2O$. Hydrogen bromide yields the analogous compound $C_2H_4Br_2O$, boiling at 148·5—151·5°. In contrast to the hydrogen bromide and iodide, the action of hydrogen chloride is very slow, as 20 grams of oxymethylene, even after six to seven days, was only partially transformed into the compound $C_2H_4Cl_2O$, an oily liquid boiling at 102—108°.

All three halogen compounds are oily liquids of an extremely pungent odour, affecting the eyes and nose; insoluble in water, but decomposed by and fuming in the air. They are soluble in ether, benzene, and acetone. Their structure is symmetrical, for the action of water corresponds quantitatively with the reaction $CH_2Br{\cdot}O{\cdot}CH_2Br + H_2O = 2CH_2O + 2HBr$, and no formic acid is produced. On heating the compound $C_2H_4Br_2O$ with water and oxymethylene at 140°, methyl bromide and formic acid were formed, from which the author concludes that the formation of formic acid and methyl haloïds (monohaloïd derivatives of methylene) on heating oxymethylene with aqueous halogen acids, is due not to oxidation or to reduction of 2 mols. of the oxymethylene, but to the reaction between 1 mol. of oxymethylene and 1 mol. of a haloid hydrin of methylene glycol, the last being the product of the reaction of the halogen hydride on oxymethylene. B. B.

Action of Halogens on Oxymethylene. By W. TISHTCHENKO (*J. Russ. Chem. Soc.*, 1887, 479—483).—The action of chlorine on oxymethylene is very simple, consisting in the substitution of chlorine for hydrogen. A large flask was used, and it was found that the temperature had no perceptible influence. In direct sunlight, however, the reaction is very rapid, and is represented by the equation $CH_2O + 2Cl_2 = COCl_2 + 2HCl$. The action of bromine is not quite so simple. It was best effected in sealed tubes at 100°. Carbonic oxide and anhydride escaped after opening the tube, and a liquid remained, which consisted of the bromide $CH_2Br{\cdot}O{\cdot}CH_2Br$, methyl bromide, hydrobromic acid and formic acid. The action of iodine on oxymethylene at 125° is analogous. B. B.

Action of Zinc Organic Compounds on Oxymethylene: Synthesis of Primary Alcohols. By W. TISHTCHENKO (*J. Russ. Chem. Soc.*, 1887, 483—486).—Wagner has found that aldehydes with zinc organic compounds yield secondary alcohols, and as oxymethylene may be regarded as an aldehyde containing a hydrogen-atom in place of an alkyl group, the formation of primary alcohols might be expected to take place. From zinc ethide and oxymethylene, after decomposition of the product of reaction by water, propyl alcohol was obtained, and in like manner normal butyl alcohol was formed when zinc propide was used.

The following method for the preparation of oxymethylene is recommended by the author:—The mixture of air and methyl alcohol vapour must be passed over platinised asbestos containing not too much platinum, and this must be placed loosely in the tube, otherwise the alcohol would be oxidised to carbonic anhydride. The temperature of the exit part of the tube only is to be kept below red heat, the part of the tube with the asbestos where the vapour enters being heated by the heat of the reaction itself. The current of air passed through methyl alcohol should be pretty rapid, but yet so that the individual bubbles are still distinguishable. When the solution obtained is placed in an exsiccator under reduced pressure over sulphuric acid, and this acid often renewed, 13·17 per cent. of dry oxymethylene remains after several weeks. It is perfectly dry and powdery if the solution be repeatedly stirred during the drying.

B. B.

Ethylene Disulphides and Ethylene Disulphones. By H. FASBENDER (*Ber.*, **21**, 1473—1477; comp. Abstr., 1887, 462).—*Ethylidene ethylene disulphide*, $CHMe<\begin{matrix}S{\cdot}CH_2\\S{\cdot}CH_2\end{matrix}>$, is a colourless oil boiling at 172—173°. The corresponding *disulphone* crystallises from water in needles which melt at 198°, and volatilise on further heating.

Propylidene ethylene disulphide, $CHEt<\begin{matrix}S{\cdot}CH_2\\S{\cdot}CH_2\end{matrix}>$, boils at 191—192°. The *disulphone* melts at 124°.

Ethylene mercaptan also condenses with valeraldehyde, and with

chloral it forms an additive product, $C_2H_4[S{\cdot}CH(OH){\cdot}CCl_3]_2$, which crystallises from ether in shining silky leaves melting at 116°.

Benzylidene ethylene disulphide, $CHPh<^{S\cdot CH_2}_{S\cdot CH_2}>$, melts at 29°, and dissolves in concentrated sulphuric acid with a deep orange-yellow colour.

Paramethoxybenzylidene ethylene disulphide,

$$OMe{\cdot}C_6H_4{\cdot}CH<^{S\cdot CH_2}_{S\cdot CH_2}>,$$

obtained from anisaldehyde and ethylene mercaptan, forms colourless needles melting at 64—65°.

Glyoxalethylenemercaptale, $C_2H_4S_2{:}CH{\cdot}CH{:}C_2H_4S_2$, forms flat, feathery leaves which melt at 133°, and sublime without decomposition. It is readily soluble in alcohol, sparingly in ether, and insoluble in water. When carefully oxidised with nitric acid in glacial acetic acid solution, it is converted into ethylenedisulphonic acid. *Dimethylmethylene ethylene disulphide*, prepared by treating acetone and mercaptan with hydrochloric acid, is a colourless oil boiling at 171°. *Dimethylene ethylene disulphone*, $CMe_2<^{SO_2\cdot CH_2}_{SO_2\cdot CH_2}>$, is obtained by oxidising the preceding compound with potassium permanganate in acetic acid solution. It crystallises in needles which melt at 232°, and distil without decomposition.

Diphenylmethylene ethylene disulphide, $CPh_2{:}S_2C_2H_4$, crystallises from alcohol in long, broad plates melting at 106°.

Ethylenemercaptandipyruvic acid, formed by mixing the mercaptan with pyruvic acid, crystallises from benzene in the form of a white, crystalline powder which melts at 96°, and is slowly decomposed on exposure to the air.

Ethylenemercaptolepyruvic acid, $COOH{\cdot}CMe{:}S_2C_2H_4$, obtained by the condensation of the preceding compound, separates from alcohol, ether, and chloroform, in clusters of short crystals melting at 102°. The *silver* salt forms colourless needles which become violet-red on exposure to the air. The acid yields ethylidine ethylene disulphone, melting at 198° when oxidised. F. S. K.

Diethylene Tetrasulphide. By H. Fasbender (*Ber.*, **21**, 1470—1472; compare Abstr., 1887, 462).—Diethylene tetrasulphide can be obtained by the action of the halogens on ethylene mercaptan in chloroform solution; also by treating the mercaptan with concentrated sulphuric acid, or with sulphuryl chloride. Furthermore by acting on an alkaline solution of the mercaptan with hydroxylamine hydrochloride. Hydroxylamine hydrochloride acts similarly on alkaline solutions of ethyl mercaptan, phenyl mercaptan and thioresorcinol.

Diethylene tetrasulphide softens at 141°, melts at 151—152°, and is not decomposed either when heated alone at 200°, or when heated with phenol. When treated with bromine, it yields an additive product, $C_4H_8S_4Br_8$, which forms well-defined, brownish-red unstable crystals. F. S. K.

Action of Feeble Mineral Acids on Allyl Alcohol. By W. Solonina (*J. Russ. Chem. Soc.*, 1887, 302—315).—Livoff and Shishukoff have shown that the action of acids converts the unsaturated alcohol, isobutenol, C_4H_8O, into its isomeride isobutaldehyde, and that isobutylene glycol is formed at the same time as an intermediary product. It was interesting to investigate the first homologue of this series, allyl alcohol, in the same direction, especially as but little was known as to the action of acids on allyl alcohol up to this date.

One volume of allyl alcohol was heated with two volumes of hydrochloric acid (containing 10 per cent. of HCl) for about 20 hours at 100°. After the lapse of this time, the tubes were found to contain one-eighth to one-tenth of a brown oily liquid, the remainder being a brownish-yellow, aqueous liquid. On repeating the process, more of the oily liquid was obtained. The oily liquid was distilled with aqueous vapour in a current of carbonic anhydride in order to prevent the absorption of oxygen. After washing with water in the presence of marble, and drying over calcium chloride, the different fractions were investigated. Fraction 45—46° was found to be allyl chloride. Fraction 94—95° was allyl ether. Fraction 135—138° was a pale-yellow oil of a pungent odour, absorbing oxygen eagerly. It exhibited the general properties of unsaturated aldehydes; its dibromide is very unstable; its formula, $C_6H_{10}O$, was confirmed by several vapour-density determinations. The oil was then subjected to the action of a continuous current of oxygen; this was absorbed and carbonic anhydride evolved. After purification, the product was boiled with calcium carbonate, and the unaltered portion distilled off. The residue, when crystallised from dilute alcohol, gave a calcium salt, $(C_6H_9O_2)_2Ca + 4H_2O$; the silver salt is $C_6H_9O_2Ag$. Both are salts of α-methyl-β-ethacrylic acid. The hydroxylamine-derivative C_6H_4NO was prepared by the action of sodium carbonate and hydroxlamine hydrochloride in a feeble alcoholic solution. After extraction with ether and recrystallisation from light petroleum, crystals were obtained melting at 48—49°, and boiling without decomposition at 193—194°. A vapour determination confirmed the above formula.

The author thinks that the aldehyde obtained by him is the α-methyl-β-ethacraldehyde, $C_2H_5{\cdot}CH{:}CMe{\cdot}CHO$, of Lieben and Zeisel (Abstr., 1883, 570). The aqueous layer was distilled over potassium carbonate, and the distillate contained a small quantity of propaldehyde together with allyl chloride. From the remaining aqueous portion, after evaporation, filtration, &c., an oily liquid was obtained, boiling between 180° and 194°, which was found to be propylene glycol. The action of dilute sulphuric acid is analogous to that of hydrochloric acid. B. B.

Isodulcitol. By E. Fischer and J. Tafel (*Ber.*, **21**, 1657—1660; compare this vol., p. 39).—*Isodulcitolcarboxylic acid*, $C_7H_{14}O_7$, is formed when 25 grams of isodulcitol dissolved in 25 c.c. of water is heated with 7·5 c.c. of anhydrous hydrogen cyanide at 30°; after one and a half hours, the whole is cooled and kept for 12 hours at the ordinary temperature. It is again heated at 30° for some hours, diluted with water and evaporated down. The residue is then heated

with an excess of baryta-water on a water-bath, dissolved in much water, treated with carbonic anhydride, and decolorised with animal charcoal. On evaporating down, the *barium salt* separates in colourless, slender plates. The yield of pure salt is 15·5 grams. The free acid could not be isolated, as it is converted into the *lactone*, $C_7H_{12}O_6$, when the acid solution is evaporated. The latter crystallises in slender, concentrically grouped needles, melts at 168°, dissolves readily in water and alcohol, very sparingly in ether. When the barium salt is reduced with hydriodic acid and amorphous phosphorus, an oil is obtained having the properties of a fatty acid.

N. H. M.

Perseite. By MAQUENNE (*Compt. rend.*, **106**, 1235—1238).—The perseite employed was extracted from the stones of the ripe fruit of *Laurus persea*, which gave about 15 grams per kilo. No saccharose was present, but the stones contained a small proportion of reducing sugar identical in properties with invert sugar.

Pure perseite melts at 188°, and is sensibly volatile. It has the composition $C_6H_{14}O_6$, and the molecular weight as determined in aqueous solution by Raoult's method is 179. The formula corresponds with 182.

The *hexacetate*, $C_6H_8O_6(C_2H_3O)_6$, is obtained by dissolving the perseite in five times its weight of boiling acetic anhydride and a fragment of zinc chloride, and recrystallising the product from alcohol. It forms a white crystalline powder, which has a somewhat bitter taste, and is insoluble in water, but dissolves in alcohol. It melts at 119°, and volatilises at a higher temperature. The *hexabutyrate*, obtained by the action of butyric chloride, is a viscous, yellowish liquid, insoluble in water, but soluble in alcohol or ether. It boils at about 300° in a vacuum with gradual decomposition. The *hexanitrate*, $C_6H_8O_6(NO_2)_6$, is obtained by adding sulphuric acid to a cold solution of perseite in nitric acid, washing the product with water, and recrystallising from alcohol of 95°. It crystallises in light, slender needles, insoluble in water, and only slightly soluble in alcohol. It softens at about 132°, and melts distinctly at 138°. At higher temperatures, it gives off nitrogen oxides. If heated rapidly, it melts without exploding, but it detonates under the hammer, although less violently than nitromannitol or nitroinosite.

All these derivatives yield perseite when treated with alkalis. It is evident that perseite is a hexahydric alcohol, and a true isomeride of mannitol and dulcitol.

C. H. B.

Products of the Action of Red Mercuric Oxide and Baryta-water on Glucose. By A. HERZFELD (*Annalen*, **245**, 27—35).—The chief product of the action of red mercuric oxide and baryta-water on glucose is gluconic acid. Glycollic and formic acids are also formed, but the presence of glyceric and trihydroxybutyric acids could not be detected.

W. C. W.

Anilides of Glucoses and some of their Transformations. By V. SOROKIN (*J. Russ. Chem. Soc.*, 1887, 377—384).—The crystalline anilides of galactose, levulose and dextrose have been

already described by the author (Abstr., 1886, 526, 683). Formerly the author heated the glucoses with aniline to its boiling point, but now he finds that the quantity of brown decomposition products formed is much smaller if the temperature is kept at 130—135°, so that the glucose is just dissolved. Still better results are obtained in alcoholic solution. For 10 grams of pure glucose, 26 grams of aniline, and 150 c.c. of alcohol of 98 per cent. are taken, and the liquid boiled until dissolution of the glucose is complete. When half the liquid has been distilled off and the residue cooled, the anilide crystallises out, especially on the addition of 2 to 3 volumes of ether. After recrystallisation from alcohol, 80 per cent. of the theoretical quantity is obtained. Methyl alcohol may be used instead of ethyl alcohol. In order to prepare the anilide of galactose, alcohol of 90 per cent. is employed, and the operation conducted as above. The preparation of the anilide of lævulose is far more difficult, a large quantity of a syrup and only a small quantity of the crystalline anilide being obtained. The separation of the latter from the former is effected by taking advantage of its solubility in alcohol. The galactose-anilide crystallises in long triclinic prisms, lævulose-anilide in rectangular plates, dextrose-anilide is only with difficulty obtained in small needles. Pure dextrose-anilide becomes brownish on heating to 140°, and melts at 147°, solidifying to a dark, vitreous mass. Its formula, $C_{12}H_{17}NO_5$, corresponds with that of its analogues. As the aqueous solutions are easily decomposed, the specific rotatory power was determined in methyl and ethyl alcoholic solution. All the solutions were found to be *lævogyrate*, so that when the aniline-residue enters a glucose molecule, the dextrogyrate power is converted into the lævogyrate in the case of galactose and dextrose, whereas that of lævulose is increased merely. The rotatory power of lævulose-anilide in alcoholic solution increases with decreasing concentration. When to an aqueous solution containing 2 to 3 per cent. of either anilide at 0° bromine is added, until the precipitate formed is coloured red, and the filtrate is subsequently treated with silver oxide and hydrogen sulphide, the corresponding glucoses are formed, and the insoluble precipitate is found to be symmetrical tribromanilide. By the action of nitric acid, the anilides are split up into glucoses and aniline, but the former are at once oxidised. From dextrose-anilide a small quantity of saccharic acid is obtained. Galactose-anilide yields mucic acid. B. B.

Fermentation of Galactose. By B. Tollens and W. E. Stone (*Ber.*, **21**, 1572—1578).—Comparative fermentation experiments were made with galactose (very carefully purified) and dextrose, and the amount of alcohol formed in each case determined. In two experiments, the yeast was supplied with food, and in two experiments the yeast alone was employed. Galactose with food fermented more slowly but nearly as completely as dextrose. The percentages of alcohol were for galactose 45·15, for dextrose 45·59, instead of 51·11 per cent. When no food was supplied, the fermentation was incomplete after seven days, and only 11·13 and 16·60 per cent. of alcohol were obtained in each case.

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In a second series of experiments, galactose and cane-sugar were fermented in presence of yeast-food, and cane-sugar with yeast alone. The same result was obtained with galactose as before, whilst the cane-sugar with yeast alone gave only 21·76 of alcohol instead of 53·80 per cent. (compare Pasteur, *Ann. Chim. Phys.* [3], 68, 337; Fudakowski, Abstr., 1878, 776; v. Lippmann, Abstr., 1885, 41; Kiliani, *Ber.*, **13**, 1384; and Bourquelot, this vol., p. 573).

N. H. M.

Determination of the Molecular Weight of Raffinose and Formaldehyde by Raoult's Method. By B. Tollens and F. Mayer (*Ber.*, **21**, 1566—1572).—In the following determinations, aqueous solutions were used: with raffinose the numbers 544, 594, 644, and 594 were obtained, which point to the formula $C_{18}H_{32}O_{16} + 5H_2O$ (compare de Vries, this vol., p. 667).

In the case of hexamethyleneamine, the numbers were 112 and 117. These numbers are in favour of the formula $C_6H_{12}N_4$ (140) rather than the formula $C_3H_6N_2$ (70) (compare Tollens, Abstr., 1884, 988). Solutions of formaldehyde were prepared by heating oxymethylene with water at 130—150°. The following numbers were obtained: 35, 34, and 34, which show that formaldehyde has the formula CH_2O. The first two numbers were from preparations which had been kept for some days; the third preparation was examined after a few hours. The authors intend to apply the method to various carbohydrates.

N. H. M.

Acetalamine and Diacetalamine. By L. Wolff (*Ber.*, **21**, 1481—1484; compare Abstr., 1887, 464, and also Wohl, this vol., p. 443).—Acetalamine and diacetalamine are produced when chloracetal is heated at 130° with ammonia.

Acetalamine, $NH_2{\cdot}CH_2{\cdot}CH(OEt)_2$, remains in the solution when the product of reaction is shaken up with ether, and is separated by adding potassium carbonate and distilling. It is a colourless liquid, of disagreeable odour, which boils at 163°, and absorbs carbonic anhydride from the air. It is readily soluble in water, alcohol, ether, and chloroform, and is decomposed when left in contact with dilute acids. The strongly alkaline, aqueous solution, with mercuric chloride, gives a white, flocculent precipitate, and with auric chloride an oil which soon solidifies to yellow plates.

Diacetalamine, $NH[CH_2{\cdot}CH(OEt)_2]_2$, is separated by fractioning the ethereal extract. It is a colourless liquid of disagreeable odour, which boils at 258—260° with slight decomposition. It is lighter than water, mixes in all proportions with alcohol, ether, and chloroform, and is decomposed by boiling with dilute sulphuric acid. When the strongly alkaline aqueous solution is heated, the base separates to a large extent in oily drops. The platinochloride,

$$NH[CH_2{\cdot}CH(OEt)_2]_2,PtCl_4,$$

crystallises in orange-red plates melting at 121°.

F. S. K.

Diazo-derivatives of Methyluracil. By R. Behrend (*Annalen*, **245**, 213—230).—*Diazo-isonitrosomethyluracil*, $C_5H_5N_5O_4$, is deposited on the addition of sodium nitrite (2 mols.) to a solution of crude amidomethyluracil in hydrochloric acid. On recrystallisation from

hot water, it is deposited in minute prisms. It is sparingly soluble in alcohol, but dissolves freely in alkalis, and is reprecipitated from alkaline solutions by acids. It is decomposed by prolonged boiling with water, or more easily by hydrochloric acid.

The nitro-derivative, $C_5H_4N_4(NO_2)O_4$, detonates at 100°. It expels carbonic anhydride from solutions of sodium carbonate. It is decomposed by warm water. Gas is evolved, and a substance of the composition $C_3H_2N_3O_2 + H_2O$ is deposited in crystals.

On reduction with stannous chloride and hydrochloric acid, diazoisonitrosomethyluracil yields hydroxylamine and isoxanthine, $C_5H_4N_4O_2$. Monobromisoxanthine, $C_5H_3BrN_2O_2 + H_2O$, crystallises in six-sided plates. At 150° it becomes anhydrous. W. C. W.

Cyanaldehyde. By P. Chautard (*Compt. rend.*, **106**, 1167—1170).—170 grams of iodaldehyde dissolved in about twice its weight of absolute alcohol was mixed with 134 grams of dry powdered silver cyanide, and the mixture heated to boiling on a water-bath for 10 hours. The product was fractionated and dried over calcium chloride. The product is *cyanaldehyde*, a colourless, limpid, somewhat refractive liquid, with an odour recalling that of ethyl acetate. It is very volatile, and burns with a luminous but not smoky flame. It boils at 71·5°; sp. gr. at 15° = 0·881; vapour-density, 2·33. It is miscible with water, and dissolves in all proportions in alcohol, ether, chloroform, and acetone. With sodium hydrogen sulphite, it forms a crystalline compound; it reduces Fehling's solution when heated, and is decomposed by soda, potash, and ammonia at a high temperature with formation of resinous products. Hydrochloric acid also yields resinous products, together with a small quantity of crystals. Boiling nitric acid converts cyanaldehyde into cyanacetic acid.

When heated with a large excess of aniline in sealed tubes at 300° for 16 hours, and the excess of aniline distilled off, cyanaldehyde yields crystals of *cyanethylidenediphenyldiamine*, $CN{\cdot}CH_2{\cdot}CH(NHPh)_2$, which melts at 113°.

Thiocyanaldehyde is obtained in a similar manner from iodaldehyde and silver thiocyanate. It is isomeric with acetyl thiocyanate, but is distinguished by its greater stability in presence of water. It is a very dense, viscid liquid with a fœtid odour, and cannot be distilled without decomposition. C. H. B.

Acetonechloroform. By C. Willgerodt and A. Genieser (*J. pr. Chem.* [2], **37**, 361—374).—During the formation of solid acetonechloroform (Abstr., 1882, 491), a liquid isomeride is produced. This is best obtained when 300 to 350 grams of powdered potassium hydroxide is gradually added to a mixture of acetone (500 grams) and chloroform (1000 grams). The filtrate from the reaction is distilled, and the fraction 165—172° rectified. Liquid acetonechloroform boils at 170° (uncorr.), is a thick, colourless oil, stable in a sealed tube in the dark, but converted by light into a dark brown polymeride. It corrodes organic fabrics, and reddens blue litmus. In contact with water it is converted into the solid isomeride, which has no corrosive action, and is not so poisonous as the liquid; it also chars at a lower temperature.

Diphenylchloromethyl dimethyl carbinol, $CPh_2Cl{\cdot}CMe_2{\cdot}OH$, is obtained by heating a mixture of acetonechloroform, solid or liquid (1 mol.), with benzene (5 to 6 mols.), in presence of aluminium chloride (2 mols.) on the water-bath for 14 to 21 days, and subsequently distilling with steam. When redistilled it is a heavy, colourless oil, boiling about 239° (uncorr.). When less benzene is used in the reaction, *phenyldichloromethyl dimethyl carbinol*,

$$CPhCl_2{\cdot}CMe_2{\cdot}OH,$$

is obtained as colourless liquid, boiling about 217°; and when an excess of benzene and a longer time are employed, *triphenylmethyl dimethyl carbinol*, $CPh_3{\cdot}CMe_2{\cdot}OH$, is formed; it is a pale-yellow liquid, boiling about 260°.

When toluene is substituted for benzene in these reactions, a mixture of *tolyldichloromethyl dimethyl carbinol*,

$$C_6H_4Me{\cdot}CCl_2{\cdot}CMe_2{\cdot}OH,$$

and *ditolylchloromethyl dimethyl carbinol*, $CCl(C_6H_4Me)_2{\cdot}CMe_2{\cdot}OH$, is obtained. The former is a bright yellow, heavy oil, boiling at 245° (uncorr.); the latter has a darker colour, and boils at 265° (uncorr.).

When paraxylene is used, the reaction takes place more readily, and *triparaxylylmethyldimethyl carbinol*, $C(C_6H_3Me_2)_3{\cdot}CMe_2{\cdot}OH$, is obtained; it is a bright greenish oil, and boils above 300°. These six condensation-products are all characterised by an odour of mushrooms.

By treating liquid acetonechloroform with phosphorus pentachloride at 100°, carefully excluding all water, hydrogen chloride is evolved, and a clear colourless liquid may afterwards be distilled off. When washed and rectified, this boils at about 151° (uncorr.), and has the formula $C_4H_6Cl_2O$; it is insoluble in water, but soluble in organic solvents; its vapour strongly attacks the mucous membrane. When heated with dry silver oxide, oxygen is substituted for the chlorine; it is unchanged when heated with water at 240°. The behaviour of the solid isomeride with phosphorus pentachloride has been described before (Abstr., 1887, 570).

The structural formula which best expresses the above reactions of liquid acetonechloroform is $CHCl_2{\cdot}CMe_2{\cdot}OCl$. A. G. B.

α-Diketones. By H. v. Pechmann (*Ber.*, **21**, 1411—1422).—The author has abandoned the sodium hydrogen sulphite method of preparing α-diketones (this vol., p. 248), and prefers to saponify the ethyl salt of methacetoacetic acid or its homologues with dilute (3 per cent.) alkali, to treat the product with sodium nitrite and sulphuric acid, and after removal of the alcohol by distillation to add 20 times its volume of dilute sulphuric acid (15 per cent.), and distil the mixture with steam (compare *loc. cit.*). The yield is not quite so good, but the method is more simple than that previously described.

Diacetyl forms with alcohol an unstable compound, $C_4H_6O_2 + 2C_2H_6O$, which boils at 74—75°, and gives the reactions of diacetyl; it also slowly combines with water forming a crystalline compound, $3C_4H_6O_2 + 2H_2O$, insoluble in water, alcohol, and ether. The *phenylhydrazone*, $COMe{\cdot}CMe{:}N_2HPh$, crystallises from dilute alcohol or acetic acid in lustrous, yellow needles, melts at 133°, and is soluble in

benzene and ether; the *diphenylhydrazone*, $N_2HPh{:}CMe{\cdot}CMe{:}N_2HPh$, crystallises from benzene in yellow needles, melts at 242°, and is very sparingly soluble in most solvents. When warmed with ortho-toluylenediamine acetate, diacetyl is converted into *dimethyltoluquinoxaline*, $C_6H_3Me\langle\begin{matrix}N{:}CMe\\N{:}CMe\end{matrix}\rangle$, which crystallises from light petroleum in hexagonal prisms, melts at 91°, boils at 270—271° without decomposition, and is more soluble in cold than in hot water, and readily soluble in the ordinary solvents. Diacetyl also reacts with aniline, forming *diacetyldianil*, $NPh{:}CMe{\cdot}CMe{:}NPh$; this crystallises from alcohol in lustrous, sulphur-yellow scales, melts at 139°, and is readily soluble in ether, soluble in alcohol and light petroleum, but insoluble in water. When concentrated aqueous ammonia is added to an aqueous solution of diacetyl and the mixture after remaining some hours in the cold is heated at 100° for an hour, a mixture of bases is obtained, of which *trimethylglyoxaline*, $\langle\begin{matrix}CMe{:}N\\CMe{:}N\end{matrix}\rangle CHMe$, constitutes the chief portion. This base crystallises from ether and light petroleum in bitter, snow-white needles, melts at 132·5—133°, boils at 271° without decomposition, and is readily soluble in water and the ordinary solvents. It is a monacid base, and yields very soluble salts; the *hydrochloride*, $C_6H_{10}N_2,HCl$, crystallises in characteristic, snow-white tufts of needles. The base reacts readily with benzyl chloride and methyl iodide, but like other glyoxalines is not affected by nitrous acid. In aqueous solution acidified with dilute sulphuric acid, diacetyl is reduced by zinc-dust to the *pinacone*,

$$COMe{\cdot}CMe(OH){\cdot}CMe(OH){\cdot}COMe,$$

which crystallises from light petroleum in long, colourless needles, melts at 96°, is soluble in the ordinary solvents, reduces Fehling's solution in the cold, and is readily oxidised to diacetyl by ferric chloride and by mercuric chloride.

Acetylpropionyl, $COMe{\cdot}COEt$, prepared from ethylic ethacetoacetate by the method just described, is a dark yellow liquid, having a quinone-like odour, and boils at 108° with the formation of a yellow vapour. It dissolves in 15 parts of water at 15°, is readily soluble in the ordinary solvents, and forms an unstable compound with alcohol. The *phenylhydrazone*, $COEt{\cdot}CMe{:}N_2HPh$, crystallises from dilute alcohol in yellowish, prismatic needles, melts at 96—98°, and is not identical with the acetylpropionyl hydrazone obtained by Japp and Klingemann by the action of diazobenzene chloride on ethylic ethacetoacetate (Trans., 1888, 540); the *diphenylhydrazone*,

$$N_2HPh{:}CEt{\cdot}CMe{:}N_2HPh,$$

crystallises from alcohol in short, strongly refracting prisms, melts at 161—162·5°, and is more soluble than the corresponding diacetyl-derivative. Acetylpropionyl reacts with aniline, forming a compound which crystallises in needles, and melts at 136—137°; it also yields a glyoxaline when treated with ammonia, but the compound could not be separated in a pure condition.

In forming these derivatives, diacetyl and its homologue resemble

glyoxal, benzil, and the other α-diketones, but they are differentiated from these on treatment with dilute aqueous alkali, inasmuch as they do not yield homologues of glycollic acid but are converted into triketones, and finally into quinones. *Dimethylquinogen*,

$$COMe\cdot CMe{:}CH\cdot CO\cdot COMe,$$

or its aldol, is obtained by carefully adding dilute aqueous soda or a dilute solution of sodium carbonate to an aqueous solution of diacetyl until it becomes colourless, when further action is at once prevented by acidification with dilute sulphuric acid, and the triketone extracted by ether. It is a yellowish syrup which does not solidify in the cold, has a bitter taste, and dissolves in the ordinary solvents. The *triphenylhydrazone*, $C_8H_{10}(N_2HPh)_3$, is an orange-yellow crystalline powder consisting of tetragonal scales united in feather-like forms, and melts at 204—205°. Further treatment with an excess of alkali converts dimethylquinogen into paraxyloquinone. In like manner, acetylpropionyl is converted into the corresponding quinogen, which forms an oil sparingly soluble in water, and yields duroquinone (Nef, Abstr., 1886, 65) by the further action of aqueous soda. The quinone is obtained from the product by extraction with benzene at 70—80°, and crystallises from wood-spirit in long, golden-yellow needles melting at 111—112°; the yield amounts to about 10 per cent. of the diketone employed. The corresponding quinol crystallises from alcohol in lustrous needles, melts at 220°, and yields a phenylhydrazone which crystallises in white, spear-like forms, and melts at 223—225°. W. P. W.

Action of Ethyl Chlorocarbonate on Salts of Fatty and Aromatic Acids. By R. Otto and W. Otto (*Ber.*, **21**, 1516—1518; comp. Otto and Rössing, Abstr., 1885, 1231).—When sodium formate is warmed with ethyl chlorocarbonate in alcoholic solution, carbonic anhydride is evolved, and ethyl formate and formic acid are produced, together with sodium chloride and small quantities of diethyl carbonate. The quantity of formic acid produced is greater than that which would be liberated from the sodium salt by the hydrochloric acid which is formed by the action of the alcohol on the ethyl chlorocarbonate.

Sodium salts of higher members of the series behave similarly when treated with ethyl chlorocarbonate in presence of alcohol, and the more carbon the acid contains the larger the quantity of free acid produced, until finally (sodium stearate) no fatty ethereal salt is formed, but only the free acid (stearic), sodium chloride, and carbonic anhydride.

About 20 per cent. of formic acid is obtained from sodium formate, whilst sodium isovalerate yields about 50 per cent. of free acid.

Equivalent quantities of sodium benzoate and ethyl chlorocarbonate in alcoholic solution yield ethyl benzoate and benzoic anhydride as principal products, besides sodium chloride, carbonic anhydride, and very small quantities of free benzoic acid.

Sodium phenylacetate and sodium oxalate also react with ethyl chlorocarbonate in presence of alcohol. F. S. K.

Brominating Organic Acids. By C. Hell (*Ber.*, **21**, 1726—1729).—A reply to Volhard (this vol., p. 129).

Action of Ammonia on Ethylidene Oxyacetate. By A. Geuther (*Annalen*, **245**, 101—102).—Ammonia acts on ethylidene oxyacetate, forming acetamide, ammonium acetate, and aldehyde. The latter forms with ammonia the base $C_8H_{13}NO$, described by Wislicenus and Heintz (*Jahresbericht*, 1858, 347). The author represents the constitution of the oxyacetate by the formula

OAc·CHMe·O·CHMe·OAc. W. C. W.

Action of Iodoform, Methylene Iodide, and Iodine on Sodium Isobutyloxide. By A. Gorboff and A. Kessler (*J. Russ. Chem. Soc.*, 1887, 428—463).—In 1858 Boutleroff showed that sodium ethoxide and iodoform yield methylene iodide, acrylic acid, and ethyllactic acids. It was expected by the authors, that iodoform with sodium isobutoxide would yield higher homologues of acrylic acid on the one hand, and of the ethoxypropionic acids on the other. Experiments made by the authors have shown that dimethacrylic acid, $C_5H_8O_2$, is formed in this reaction, as was expected, but the reaction is very complicated with regard both to the number and the character of the products formed. The yield of methylene iodide especially varied greatly, and as it has a disturbing action, it was necessary to conduct the reaction in such a way that its production was reduced to a minimum. Sodium isobutoxide was first prepared from 200 grams of isobutyl alcohol and 20 grams of sodium, and to this 100 grams of iodoform was added after a time. When the first violent reaction was over and the volatile portion (B) had distilled off, the residue (A) was extracted with water, the isobutyl alcohol removed by distillation, tartaric acid added, and the distillation continued. From the distillate containing the organic acids, the following were separated by fractional distillation: formic acid, and isobutyric acid; the portion boiling above 180°, when distilled under reduced pressure, yielded *dimethacrylic acid*, $CMe_2{:}CH{\cdot}COOH$, which was finally obtained in prismatic crystals melting at 69·5—70°, and boiling at 194—195°, sublimation taking place at a lower temperature. Ten degrees above the boiling point, the acid decomposes into isobutylene and carbonic anhydride. The monoclinic dibromide melts at 107—108°. From the higher boiling portions, besides dimethacrylic acid, α-isobutoxyisobutyric acid, $C_4H_9{\cdot}O{\cdot}CMe_2{\cdot}COOH$, was obtained.

The oily portion from A was mixed with the oily portion obtained from the higher boiling acids, and distilled under reduced pressure, when an aromatic neutral liquid was obtained of the composition $C_8H_{14}O_2$, and having the properties of an octolactone. This the authors consider to be isomeric with the octolactones of Young and of Emmert and Friedrich. The portion B has an ethereal odour, and contains, besides isobutyl alcohol, isobutylene, methyl isobutyl ether, and diisobutyl methylene ether, a compound of the composition $C_{12}H_{26}O_3$, and a mixture of substances which could not be separated by fractional distillation under reduced pressure. The compound

$C_{12}H_{26}O_3$ yielded isobutyl iodide when heated with hydriodic acid, showing that it contains isobutoxyl groups, and with acetic acid α-hydroxyisobutaldehyde was obtained as a product of decomposition, together with isobutyl acetate; of the last, according to quantitative experiments, 2 mols. are formed, and the authors therefore consider the compound $C_{12}H_{26}O_3$ to be the isobutyl alcoholate of α-isobutoxyisobutaldehyde, $C_4H_9{\cdot}O{\cdot}CMe_2{\cdot}CH(OH){\cdot}O{\cdot}C_4H_9$, which, as quantitative experiments show, decomposes into, $C_4H_{10}O$ and $C_4H_9{\cdot}O{\cdot}CMe_2{\cdot}COH$. Of the products obtained in this reaction, the following are derived from the iodoform: acetic and dimethacrylic acids, methyl isobutyl ether and diisobutyl methylene ether. In the case of the formation of isobutyric and isobutyl-α-hydroxyisobutyric acids, of the octolactone and of the compound $C_{12}H_{26}O_3$, the carbon-atom of iodoform could not have taken any part, as they contain a number of carbon-atoms which corresponds with single or multiple isobutylic groups. No isobutyl orthoformate is formed, as might have been expected, for iodoform acts on isobutyl alcohol in such a way that, in the first place, reduction of the iodoform to methylene diiodide takes place, which with sodium isobutoxide yields diisobutyl methylene ether, and then a further reduction to methyl iodide which with an excess of the alkoxide yields methyl isobutyl ether. Direct experiments have shown that the said ethers, together with isobutyric acid and the octolactone, are obtained when sodium isobutoxide acts on methylene diiodide. The hydrogen which had reduced the triiodide to the diiodide and moniodide, must have been taken from the alcohol, just as in the experiments of Bolas and Groves on carbon tetrabromide, which with alcohol yielded bromoform together with aldehyde and hydrogen bromide. The authors give a theoretical explanation of the formation of the compound $C_{12}H_{26}O_3$.

On acting with free iodine on sodium isobutoxide, the compound $C_{12}H_{26}O_3$ was obtained, together with octolactone and α-isobutoxyisobutyric acid. It seems that by acting with iodine (and bromine) on the metallic derivatives of other monatomic alcohols, substances analogous to those obtained above are formed in some cases. The authors propose to investigate this matter. B. B.

Isoleïc Acid. By M. C. and A. Saytzeff (*J. pr. Chem.* [2] **37**, 269—290; compare Abstr., 1887, 30).—When iodostearic acid, obtained by the action of hydriodic acid on ordinary oleïc acid, is heated in a reflux apparatus with alcoholic potash, and the product of the reaction is decomposed with sulphuric acid, together with ordinary oleïc acid, an isomeride is formed which melts and solidifies at 44—45°. This solid oleïc acid may be also prepared by distilling hydroxystearic acid, obtained by the successive action of sulphuric acid and water on ordinary oleïc acid. The best yield is obtained if the operation is conducted under a pressure of 100 to 150 mm., when the distillation takes place at 280—300°. Oleïc and isoleïc acids can be separated by means of their zinc salts, that of the latter being less soluble in alcohol.

Isoleïc acid, $C_{18}H_{34}O_2$, is insoluble in water, dissolves readily in alcohol and ether, and forms a liquid dibromide, $C_{18}H_{34}O_2Br_2$, isomeric

with that obtained from oleïc acid, but differing from it in not reacting in the cold with silver oxide. It furnishes a *dihydroxystearic* acid which differs from its isomeride, not only in its melting point (76—78°) and solidifying point (66—64°), but also in being much more soluble in alcohol and ether. This new dihydroxystearic acid on treatment first with hydriodic acid, and then with tin and hydrochloric acid, gives ordinary stearic acid.

Isoleïc acid combines with hydriodic acid to form a new iodostearic acid. This is a thick yellow oil, and differs from its isomeride in not readily reacting with silver oxide, in being more soluble in alcohol and ether, and in undergoing no change on distillation.

Iodostearic acid, prepared from isoleïc acid, gives only isoleïc acid when boiled with alcoholic potash, whilst that formed from ordinary oleïc acid gives a mixture of oleïc and isoleïc acids. When fused with potash, isoleïc acid behaves like its isomerides, and gives a mixture of palmitic and acetic acids. When oxidised with permanganate in alkaline solution, isoleïc acid yields a mixture of dihydroxystearic acid and a saturated bibasic acid not yet investigated.

Two hydroxystearic acids are formed when isoleïc acid is treated first with sulphuric acid and then with water. Of these, one is identical with that prepared from the iodostearic acid derived from ordinary oleïc acid, the other with that derived from the iodostearic acid prepared from isoleïc acid. The behaviour of the two isomerides leads the authors to give the formula $CH_3\cdot[CH_2]_{13}\cdot CH_2\cdot CH{:}CH\cdot COOH$, to isoleïc acid, ordinary oleïc acid being represented by the formula $CH_3\cdot[CH_2]_{13}\cdot CH{:}CH\cdot CH_2\cdot COOH$. G. T. M.

Acids from Drying Oils. By K. Hazura (*Monatsh.*, **9**, 180—197; compare Abstr., 1887, 359, 798, 913).—*Isolinusic acid* (15·8 per cent.), and dihydroxystearic acid (1·2 per cent.) are obtained together with sativic acid (6·5 per cent.) and linusic acid (4·5 per cent.) when linoleïc acid is oxidised with alkaline potassium permanganate (Abstr., 1887, 798).

Isolinusic acid, $C_{18}H_{36}O_8$, crystallises in small, anhydrous, prismatic needles which melt at 173—175°, are insoluble in ether, benzene, toluene, carbon bisulphide and chloroform, sparingly soluble in cold water and cold alcohol, but readily soluble in hot water or hot alcohol. *Acetylisolinusic* acid has the same properties as acetyllinusic, but it is more sparingly soluble in ether. The analysis of this acetyl-derivative showed that six hydroxyl-groups and one carboxyl-group are present in isolinusic acid, the formation of which from the liquid fatty acid of linseed oil necessitates the assumption that the glyceride of an acid, $C_{18}H_{30}O_2$, isomeric with linolenic acid, is also present in linseed oil. The author names this acid *isolinolenic acid*.

The alkaline salts of isolinusic acid are very readily soluble in water; the *potassium* and *sodium* salts crystallise from water in microscopic needles. The *barium* salt crystallises from hot water in microscopic needles. The *calcium* salt is soluble in hot water. Copper sulphate produces a green, silver nitrate, a white precipitate in an aqueous solution of the alkaline salts; both precipitates are insoluble in water.

Sativic acid crystallises either in long, prismatic, microscopic needles, or in pyramidal prisms according to the purity and concentration of the solution. It melts at 173°.

Linusic acid (Abstr., 1887, 798), crystallises from dilute aqueous solutions in needles, but from concentrated solutions in rhombic plates, the opposite ends of which are often blunt. When sativic acid is treated with nascent hydrogen iodide, a thick, brownish, oily iodo-compound is obtained, and this oil is converted into stearic acid by nascent hydrogen. Sativic acid is therefore tetrahydroxystearic acid.

From the regular behaviour of alkaline solutions of unsaturated fatty acids towards potassium permanganate (Abstr., 1887, 913), it follows that the liquid fatty acid from linseed oil consists of oleïc acid, linoleïc acid, linolenic acid, and isolinolenic acid.

Oleïc acid dibromide, linoleïc acid tetrabromide, linolenic acid hexabromide, and isolinolenic acid hexabromide, are formed when linoleïc acid is treated with bromine. F. S. K.

Acids from Drying Oils. By K. Hazura and A. Grüssner (*Monatsh.*, **9**, 198—207).—From the author's experiments, based on the regular behaviour of unsaturated fatty acids towards alkaline permanganate (Abstr., 1887, 913), it follows that the fatty acids from hemp-seed oil, poppy oil, nut oil, and cotton-seed oil, are not homogeneous compounds, but mixtures of unsaturated acids.

The fatty acids from hemp-seed and nut oil consist chiefly of linoleïc acid, together with small quantities of linolenic, isolinolenic, (compare preceding Abstract), and oleïc acid.

The fatty acid from poppy oil consists chiefly of linoleïc acid, together with considerable quantities of oleïc acid, and very small quantities of linolenic and isolinolenic acids.

The fatty acid from cotton-seed oil contains only linoleïc acid and oleïc acid.

The method employed in determining the constitution of the fatty acid was, in all cases, the following:—It is oxidised with alkaline potassium permanganate, the product (A + B) precipitated with sulphuric acid, dried, and treated with cold ether; the dihydroxystearic acid (A) dissolves, and the residue (B) is boiled out with water to separate the sativic acid from any dihydroxystearic acid which had not been dissolved by the ether. The acids thus separated were recognised by the usual means. The filtrate from the mixture (A + B) is neutralised with soda, evaporated, again precipitated with sulphuric acid, and the precipitate extracted with ether; the residue is recrystallised from alcohol or water, and the acid mixture (linusic and isolinusic acids) examined microscopically. In the case of cotton-seed oil, the precipitate, which probably contained azelaïc acid only, was completely soluble in ether.

F. S. K.

Halogen Substitution Products of Ethyl Acetoacetate and their Behaviour with Sodium Ethoxide. By W. Mewes (*Annalen*, **245**, 58—84).—Ethyl dichloracetoacetate is formed by

saturating ethyl acetoacetate with chlorine in diffused daylight. If the product is treated with chlorine in direct sunlight, the trichloro- and tetrachloro-derivatives are formed.

Chlorobromo-substitution products are obtained by the action of bromine on the chloro-substitution products, and also by the action of chlorine on the bromo-derivatives.

Sodium ethoxide decomposes ethyl chloracetoacetate, forming ethyl acetate and chloracetate. Ethyl dichloracetoacetate is split up into ethyl acetate and dichloracetate. The chief product of the action of sodium ethoxide on ethyl trichloracetoacetate is ethyl dichloracetate. Ethyl acetate and chloracetate are also formed in small quantity. Under similar treatment, ethyl tetrachloracetoacetate yields ethyl acetate, chloracetate, and dichloracetate.

Ethyl bromacetoacetate gives a good yield of ethyl succinosuccinate, when it is treated with sodium ethoxide. The author confirms Wedel's statement (Abstr., 1884, 434) that the chief product of the action of sodium ethoxide on ethyl dibromacetoacetate is ethyl quinonehydrodicarboxylate. When the chlorobromo-substitution products of ethyl acetoacetate are treated with sodium ethoxide, sodium bromide is invariably formed; thus ethyl monochlorodibromacetoacetate yields ethyl chlorobromacetate. W. C. W.

Homologues of Ethyl Acetocyanacetate. By A. HALLER (*Compt. rend.*, **106**, 1083—1085).—The homologues of ethyl acetocyanacetate are obtained by the action of the corresponding acid chlorides on ethyl sodiocyanacetate. 22·6 grams of ethyl cyanacetate dissolved in 30 grams of absolute alcohol is mixed with a solution of 4·6 grams of sodium in 100 parts of absolute alcohol, and the theoretical quantity of the acid chloride, diluted with four or five times its volume of ether, is added gradually to the well-cooled mixture. The product is isolated in the manner previously described (Abstr., 1887, 1029).

The compounds thus obtained are colourless, strongly acid liquids, with an odour resembling both that of the ethyl cyanacetate and that of the corresponding acid. Their salts are usually soluble in alcohol, from which they often separate in viscous masses which will not crystallise. They can, however, be obtained in crystals by dissolving them in dilute alcohol and evaporating spontaneously over sulphuric acid.

Ethyl propiocyanacetate, $CH_3Me{\cdot}CO{\cdot}CH(CN){\cdot}COOEt$, boils at 155—165°, under a pressure of 50 mm. The calcium salt crystallises in long needles, which contain 2 mols. H_2O, and are very soluble in water and alcohol.

Ethyl butyrocyanacetate, $CH_3Me{\cdot}CH_2{\cdot}CO{\cdot}CH(CN){\cdot}COOEt$, boils at 166—178° under a pressure of 66 mm. The calcium salt forms crystalline crusts which contain 2 mols. H_2O; the barium salt crystallises in nacreous mamelons which contain 3·5 mols. H_2O. The ferric salt is a viscous, non-crystalline, deep-red compound, soluble in ether.

Ethyl isobutyrocyanacetate, $CHMe_2{\cdot}CO{\cdot}CH(CN){\cdot}COOEt$, boils at 170—177°, under a pressure of 85 mm. The calcium salt forms

distinct crystals which contain 2 mols. H_2O and are slightly soluble in water, and soluble in alcohol. C. H. B.

Action of Acetic Anhydride on Levulinic Acid. By. G. MAGNANINI (*Ber.*, **21**, 1523—1524).—An *acid*, $C_9H_{10}O_4$, is obtained by heating levulinic acid at 200—225° with acetic anhydride. It crystallises from boiling water in small needles melting at 151·5—152°. The *silver* salt, $C_9H_9O_4Ag$, is a crystalline compound, which is sparingly soluble in water and remains unchanged on exposure to the air. The *barium* salt, $(C_9H_9O_4)_2Ba + H_2O$, is also crystalline. F. S. K.

Methyl Propiopropionate. By C. PINGEL (*Annalen*, **245**, 84—95).—Methyl propiopropionate boils at 187° (corr.), and its sp. gr. at 20° is 1·070. It is converted into a mixture of an oily and a crystalline amide by treatment with strong ammonia. The oily liquid has the composition $C_7H_{13}NO_2$, and the crystalline amide is probably $C_7H_{13}NO_2 + C_5H_9NO_2$. Diethyl ketone and methyl ethyl ketone are formed by the action of sodium hydroxide on methyl propiopropionate.

Methyl methylpropiopropionate and methyl ethylpropiopropionate are respectively formed by the action of methyl or ethyl iodide on methyl propiopropionate and sodium ethoxide in sealed tubes at 100°. The methyl salts of methylpropiopropionic, ethylpropiopropionic, propylpropiopropionic, and isobutylpropiopropionic acids boil at 193—196°, 208°, 217—221°, and 230—231° respectively. W. C. W.

New Synthesis of Dihydric Monobasic Acids from Ketones. By S. REFORMATSKY (*J. Russ. Chem. Soc.*, 1887, 298—299).—By the action of zinc and allyl iodide on ethyl chloracetate a compound is obtained which contains oxygen, is neutral and unsaturated, and decomposes on distillation. It is insoluble in water, and very stable, not being decomposed by alkalis or acids. It has not the properties of an alcohol. It would seem that it is formed by the mutual reaction of 2 molecules of ethyl chloracetate in the presence of zinc. Acetone with ethyl chloracetate and zinc, or better, ethyl iodacetate and zinc, yields β-dimethylethylenelactic (hydroxyvaleric) acid. The research is being continued. B. B.

Action of Aniline on Isosaccharin. By B. SOROKIN (*J. pr. Chem.* [2], **37**, 318—320).—When aniline (3 parts) is heated to the boiling point with isosaccharin (1 part) in an oil-bath, and ether is added to the product, a substance of a brownish colour is precipitated, which on crystallisation from alcohol forms colourless needles, having the formula $C_{12}H_{17}NO_5$, and melting at 165° without decomposition. This substance, which is formed by the direct union of 1 mol. of aniline with 1 mol. of isosaccharin, without elimination of water, is readily dissolved by alcohol and water, but the solution is lævorotatory. An aqueous solution is slightly acid; it does not, however, decompose calcium carbonate, but on treatment with lime-water forms calcium isosaccharinate, $(C_6H_{11}O_6)_2Ca$, and aniline. The substance is an anilide of isosaccharinic acid of the formula $C_5H_{11}O_4{\cdot}CO{\cdot}NHPh$, and

like the anilides of other acids readily splits up into its components on treatment with acids and alkalis. G. T. M.

Preparation of Dibromosuccinic Acid. By J. Gorodetzky and C. Hell (*Ber.*, **21**, 1729—1733).—Dibromosuccinic acid is best prepared by heating a mixture of 30 grams of dry succinic acid, 0·15 gram of amorphous phosphorous, and 21 c.c. of dry bromine, in three equal parts in sealed tubes at 120—130° for about six hours. 46 gram of dibromosuccinic acid, 12·5 grams of monobromosuccinic acid, and 2·5 grams of unchanged succinic acid are thus obtained. The product is well rubbed several times with lukewarm water, and the residual dibromosuccinic acid crystallised quickly from boiling water. When the temperature is quickly raised to 140°, and then lowered gradually to 130—120°, the risk of bursting the tubes is considerably lessened. F. S. K.

Action of Ethyl Iodide and Zinc, and of Allyl Iodide and Zinc on Ethereal Malonates. By B. Martinoff and S. Shukoffsky (*J. Russ. Chem. Soc.*, 1887, 297—298).—In the first reaction, ethereal salts of diethylmalonic acid are formed, and in the second corresponding salts of diallylmalonic acid. Hydrocarbons are produced at the same time. B. B.

Crystallised Saccharic Acid. By O. Sohst and B. Tollens (*Annalen*, **245**, 1—27).—Saccharic acid is best prepared by adding 100 grams of powdered potato-starch suspended in 100 c.c. of water to 500 c.c. of nitric acid, sp. gr. 1·15. The mixture is heated at 60—70° until the evolution of red fumes almost ceases, and the product is then diluted with an equal volume of water and the warm liquid neutralised by potassium carbonate. On the addition of strong acetic acid, the acid potassium salt, $C_6H_9O_8K$, is deposited in microscopic crystals. This salt is converted into silver saccharate by double decomposition, and the free acid is obtained by acting on the silver salt with hydrochloric acid. On leaving the concentrated solution for some days in an atmosphere free from ammonia, crystals of saccharic acid are deposited. After drying on porous plates at the ordinary temperature, the crystals have the composition $C_6H_8O_7$, the anhydride of saccharic acid. At the ordinary temperature, saccharic acid is monobasic, but the hot aqueous solution neutralises two equivalents of alkali. Saccharic acid behaves like a lactonic acid. Its constitution is probably

$$OH\cdot CH<\begin{matrix}CH(OH)\\CO\text{—}O\end{matrix}>CH\cdot CH(OH)\cdot COOH.$$

The specific rotation of a freshly prepared aqueous solution of the crystallised acid is + 37·6—37·9°, but after a time it steadily diminishes to +18·9—22·5°. On the other hand, a solution prepared by the addition of hydrochloric acid to the ammonium salt only exhibits a rotatory power of +8·4—9·3°, which rises to +22·66° in course of time. The saccharates of ammonium, $C_6H_9O_8\cdot NH_4$, and magnesium, $C_6H_8O_8Mg + 3H_2O$, are crystalline. Barium forms two normal

saccharates, an anhydrous crystalline salt, and an amorphous salt containing $2\frac{3}{4}$ or 3 mols. H_2O. At 150°, saccharic acid is decomposed by strong hydrochloric acid, yielding dihydromucic acid.

W. C. W.

Metasaccharic Acid. By H. Kiliani (*Ber.*, **21**, 1422—1424).—Maquenne (this vol., p. 677) has recently stated that the diphenylhydrazone of saccharic acid melts at the same temperature and has properties similar to those of the diphenylhydrazone of metasaccharic acid (*ibid.*, p. 46), and has suggested that the two acids may be identical. The author, however, points out that the diamides of the two acids are not identical, and that potassium metasaccharate, unlike the saccharate, does not yield the potassium hydrogen salt on treatment with acetic acid. Dextrose and gluconic acid, when oxidised under the same conditions as arabinosecarboxylic acid, are not converted into the double lactone of metasaccharic acid, and it was not found possible to obtain saccharic acid by heating the double lactone with water, as the latter almost completely decomposed. W. P. W.

Alkyl Salts of Sulphoacetic Acid and Ethylidenedisulphonic Acid. By R. Mauzelius (*Ber.*, **21**, 1550—1552).—*Diethylsulphoacetate*, $SO_3Et{\cdot}CH_2{\cdot}COOEt$, is prepared by the action of ethyl iodide on the silver sulphonate of ethyl acetate. The product is extracted with ether and dried with calcium chloride; on distilling off the ether, a yellowish liquid is obtained which can be made colourless by heating it in a beaker. It does not distil without decomposition.

Diethyl ethylidenedisulphonate, $CHMe(SO_3Et)_2$, is formed when the corresponding silver salt is gently heated with ethyl iodide. It is a slightly red oil of peculiar odour, readily soluble in alcohol and ether, insoluble in aqueous soda and in water. It decomposes readily when heated. The *silver salt*, $CHMe(SO_3Ag)_2 + H_2O$, crystallises in slender needles. The *sodium-derivative*, $CMeNa(SO_3Et)_2$, prepared by the action of sodium ethoxide on the ethyl salt, forms globular groups of small needles.

Butanedisulphonic acid, $CMeEt(SO_3H)_2$, is formed when the above sodium-derivative is boiled with alcohol and ethyl iodide in a reflux apparatus. The product is dissolved in water and neutralised, when the *sodium salt* separates in long, flat needles, very readily soluble in water. N. H. M.

Action of Iodobenzene on Silver Nitrite. By A. Geuther (*Annalen*, **245**, 99—101).—Silver nitrite does not act on iodobenzene at a temperature below 135°, but between 140° and 150° a reaction takes place resulting in the formation of trinitrophenol, silver iodide, and metallic silver. W. C. W.

Additive Product of Tribromodinitrobenzene and Tetrabromodinitrobenzene. By C. L. Jackson and G. D. Moore (*Ber.*, **21**, 1707—1708).—An additive product, $2C_6HBr_3(NO_2)_2,C_6Br_4(NO_2)_2$, is formed when tribromodinitrobenzene (2 mols.) and tetrabromodinitrobenzene (1 mol.) are mixed together in benzene solution. It crystallises from alcohol in rhombic leaves melting at 165°, which are

tolerably stable and can be recrystallised from ordinary solvents, but are partially decomposed by methyl alcohol or by a mixture of methyl and ethyl alcohol. F. S. K.

Desmotropy in Phenols. Bisecondary Pentethylphloroglucinol. By J. Herzig and S. Zeisel (*Monatsh.*, **9**, 217—226).—*Pentethylphloroglucinol*,

$$C_6O_2Et_5(OH)\ [OH : Et_2 : O : Et_2 : O : Et = 1 : 2 : 3 : 4 : 5 : 6],$$

is prepared as follows: phloroglucinol (1 mol.) is mixed with potash (3 mols.), dissolved in lukewarm 25 per cent. alcohol, and ethyl iodide (3 mols.) added. After the first somewhat energetic reaction ceases, the mixture is warmed for two hours, the alcohol distilled off, and the residue dissolved in water and extracted with ether. The ethereal solution is then shaken with potash until the latter is no longer coloured yellow, and on evaporating the ether a yellow, indifferent oil (Benedikt, this Journal, 1876, i, 250; Will and Albrecht, Abstr., 1884, 1335) remains. The potash is neutralised with hydrochloric acid with addition of a little sodium sulphite, and extracted with ether; when the ether is distilled, a brown product remains which is again treated with potash and ethyl iodide as described above, and the products separated as before. A yellow, indifferent oil and a compound which is soluble in potash are again obtained. The potash solution is acidified, extracted with ether, and the ether distilled, when the greater part of the residual pentethylphloroglucinol quickly solidifies. It crystallises from alcohol in white plates melting at 91—94°, but when the alcohol is too dilute, it separates in an oily condition and then solidifies to leafy crystals. It is not acted on when boiled with hydrogen iodide for two hours, and neither this compound nor the indifferent oil shows the pinewood reaction of phloroglucinol.

Hexethylphloroglucinol, $C_6O_3Et_6$, is obtained by treating the penta-derivative with potash and ethyl iodide, but even with a large excess of the reagents a considerable quantity of pentethylphloroglucinol remains unchanged. It is an oily compound and completely insoluble in potash. One ethyl-group is readily and quantitatively eliminated as ethyl iodide, when it is treated with hydrogen iodide according to Zeisel's method.

The indifferent oil alluded to above is probably a mixture of the ethyl-derivatives of various pseudo-forms of phloroglucinol.

F. S. K.

Preparation of Nitramines from Nitrophenols. By A. Barr (*Ber.*, **21**, 1541—1549).—Merz and Ris (Abstr., 1886, 872) obtained ortho- and para-nitraniline by heating the corresponding nitrophenols with aqueous ammonia. In a similar manner Witt (Abstr., 1886, 947) prepared dinitronaphthylamine from α-dinitronaphthol.

Dinitraniline, $[NH_2 : (NO_2)_2 = 1 : 2 : 4]$, is obtained by heating 3 grams of dinitrophenol (m. p. 112°) with 10 c.c. of 27 per cent. ammonia for 16 hours at 175°; the product is filtered and the red needles shaken with water containing ammonia and ether. The ethereal solution is separated, dried, and the ether distilled off. It

crystallises in light-yellow crystals melting at 176° (not 182°). The yield is 60 per cent. of the weight of dinitrophenol.

Metanitroparatoluidine is prepared by heating 3 grams of pure nitrocresol with 8 c.c. of 35 per cent. ammonia for 16 hours at 170—180°. The yield is 63 per cent. of the weight of the nitrocresol.

Dinitrotoluidine, [Me : NH_2 : $(NO_2)_2$ = 1 : 2 : 3 : 5], is formed when 3 grams of dinitrocresol is heated with 10 c.c. of 27 per cent. ammonia for 20 hours at 175—185°. It crystallises from xylene in yellow prisms melting at 208°.

Dinitroisobutylaniline, $NH_2{\cdot}C_6H_2(C_4H_9)(NO_2)_2$, prepared by heating 3 grams of the dinitrophenol with 10 c.c. of 27 per cent. ammonia for 15 hours at 175°, crystallises from warm alcohol in slightly yellowish-brown, feathery crystals melting at 127°. It sublimes in yellow needles, is readily soluble in ether and warm alcohol, insoluble in boiling water.

Dinitrophenylenediamine, [$NH_2 : NO_2 : NH_2 : NO_2$ = 1 : 2 : 3 : 4], is obtained when 4 grams of dinitroresorcinol is heated with 15 c.c. of 27 per cent. ammonia for 20 hours at 115°. It crystallises from glacial acetic acid in groups of light yellowish-brown needles which melt with decomposition at about 250°. It is very sparingly soluble in alcohol, ether, and benzene. The isomeric dinitro-amido-compound described by Nietzki and Hagenbach melts at about 300° (Abstr., 1887, 477).

Trinitrophenylenediamine, $C_6H_5N_5O_6$, obtained by boiling diethyltrinitroresorcinol with alcoholic ammonia for a short time, separates from its solution in glacial acetic acid in crystalline grains which do not melt at 250°, and detonate slightly when heated in a flame. It is almost insoluble in the usual solvents except glacial acetic acid. Boiling aqueous sodium carbonate decomposes it with liberation of ammonia. When reduced with tin and strong hydrochloric acid, *pentamidobenzene hydrochloride*, $C_6H(NH_2)_5{,}3HCl$, is formed. This crystallises in hair-like needles, very readily soluble in cold water, almost insoluble in alcohol and ether. When the aqueous solution is exposed to air, it becomes violet and a dark substance separates. The free base was not isolated. The *pentacetyl-derivative*, $C_6H(NHAc)_5$, forms an almost insoluble, amorphous powder. Pentamidobenzene reacts without difficulty with benzaldehyde, phenanthraquinone, and benzil, &c., with formation of crystalline compounds.

Trinitrophenylenediamine could not be prepared from trinitroresorcinol and ammonia. N. H. M.

Cyanacetates of the Benzene Series. By A. Haller (*Compt. rend.*, **106**, 1171—1174).—An aqueous solution of sodium nitrite containing 1 gram-molecule per litre, and a solution containing 1 gram-molecule of aniline and 3 gram-molecules of hydrochloric acid per litre are prepared, and likewise hydrochloric acid solutions of orthotoluidine and paratoluidine half the strength of the aniline solution.

100 c.c. of the aniline solution or 200 c.c. of toluidine solution is cooled at 0° and mixed with 100 c.c. of the nitrite solution and a solution of ethyl sodiocyanacetate obtained by treating 11·3 grams of

ethyl cyanacetate in 50 grams of ethyl alcohol with 2·3 grams of sodium dissolved in 50 grams of alcohol. If methyl-derivatives are required, 9·9 grams of methyl cyanacetate in 30 grams of methyl alcohol is substituted for the ethyl cyanacetate and sodium methoxide for the ethoxide. The product separates as a yellow precipitate which is dissolved in potash, precipitated with sulphuric acid, washed with water, dried, and recrystallised from alcohol.

The derivatives thus obtained are yellow crystalline compounds insoluble in water, slightly soluble in cold alcohol, soluble in hot alcohol and in benzene, chloroform, ether, and alkalis. They also dissolve in hot solutions of alkaline carbonates.

Methyl azobenzenecyanacetate forms slender needles which melt at 86·5°. The corresponding ethyl salt melts at 124·9°. *Methyl azotoluenecyanacetate* [1 : 2] forms silky needles which melt at 167·2°. The ethyl salt melts at 125·8°. *Methyl azotoluenecyanacetate* [1 : 4] melts at 133 5°, and the ethyl salt at 74·4°.

The solubility in alkalis indicates that these compounds have the constitution $R{\cdot}N_2{\cdot}CH(CN){\cdot}COOR$, and not $R{\cdot}NH{\cdot}N{\cdot}C(CN){\cdot}COOR$, and the solubility in alkaline carbonates shows that the introduction of N_2 into the CH_2-group of the cyanacetates has greatly increased the activity of the acid function. C. H. B.

Bromacetorthotoluide and some of its Derivatives. By P. W. Abenius and O. Widman (*Ber.*, **21**, 1662—1664).—*Bromacetorthotoluide*, prepared by the action of bromacetic bromide on orthotoluidine dissolved in benzene, crystallises in white needles melting at 113°. When this compound or the chlorinated derivative (m. p. 111—112°) is boiled with aqueous potash, *diorthotolyldiketopiperazine*, $C_6H_4Me{\cdot}N<\begin{matrix}CO{\cdot}CH_2\\CH_2{\cdot}CO\end{matrix}>N{\cdot}C_6H_4Me$, is obtained. This crystallises in well-formed plates which melt at 159—169°, is insoluble in bases and in dilute acids. The platinochloride (with 4 mols. H_2O) melts at 176°. When boiled with an excess of alcoholic potash for half an hour, *orthotolylglycinylorthotolylglycine* (*orthotolylglycinorthotolylamidoacetic acid*), $C_7H_7{\cdot}NH{\cdot}CH_2{\cdot}CO{\cdot}N(C_7H_7){\cdot}CH_2{\cdot}COOH$, is formed; this melts at 129°. Phosphorus pentachloride reacts with ditolyldiketopiperazine at 140° with formation of the compound

$$\begin{matrix}C_7H_7{\cdot}N—CCl{\cdot}CO\\ \quad|\quad|\quad|\\ CO{\cdot}CCl{\cdot}N{\cdot}C_7H_7\end{matrix},$$

which crystallises in slender needles melting at 200—201°.

Chloracetylorthotolylglycine, $C_6H_4Me{\cdot}N(CO{\cdot}CH_2Cl){\cdot}CH_2{\cdot}COOH$, is prepared by mixing ethereal solutions of chloracetyl chloride and orthotolylglycine; it crystallises from benzene in well-formed plates and melts at 116—117°; when heated with 2 mols. orthotoluidine at 160°, it is converted into diorthotolyldiketopiperazine.

Tetrabromodiorthotolyldiketopiperazine, $C_{18}H_{14}Br_4O_2$, is obtained by brominating orthacetotoluide at 160° and boiling the orthobromacetodibromotoluide so formed with alcoholic potash. It crystallises in plates and melts at 277°. N. H. M.

Lactones derived from Glycines. By P. W. Abenius (*Ber.*, **21**, 1668—1669).—*Glycolylorthotolylglycin*,

$$COOH{\cdot}CH_2{\cdot}N(C_7H_7){\cdot}CO{\cdot}CH_2{\cdot}OH,$$

is formed when chloracetylorthotolylglycin (preceding Abstract) is boiled with an excess of soda. It crystallises in hexagonal leaves or prisms which melt at 143—144°, are readily soluble in water, alcohol, ether, chloroform, acetone, &c., but tolerably sparingly soluble in benzene.

Anhydroglycolylorthotolylglycin, $C_7H_7{\cdot}N<\begin{matrix}CH_2{\cdot}CO\\CO{\cdot}CH_2\end{matrix}>O$, is obtained by heating the preceding compound at 160°. It crystallises from alcohol, in which it is readily soluble, in long prisms melting at 108—109°. It behaves like a lactone, dissolves slowly in cold caustic alkalis and in warm alkaline carbonates with regeneration of glycolylorthotolylglycin. F. S. K.

Pentamidobenzene. By A. W. Palmer and C. L. Jackson (*Ber.*, **21**, 1706—1707).—Triamidodinitrobenzene, prepared from tribromodinitrobenzene melting at 192°, is an amorphous, yellow compound. It yields a colourless reduction product, the hydrochloride of which, $C_6H(NH_2)_2(NH_3Cl)_3$ or $C_6H(NH)_2(NH_3Cl)_3$, is a white powder which gradually turns brown on exposure to the air. F. S. K.

Orthazotoluene. By W. Pospechoff (*J. Russ. Chem. Soc.*, 1887, 402—413).—According to Werigo and Petrieff this compound melts at 137°. The "ortho-azotoluene" of Hoogewerff and van Dorp melts at 55°, and is different from Petrieff's. Kissel, on repeating Hoogewerff and van Dorp's experiment, obtained a compound melting at 137°. Schultz, without regard to any existing discrepancies, gives the melting point as 55°. In order to settle the question, the author has reinvestigated the different processes by which orthoazotoluene may be formed. By the action of sodium amalgam on nitrotoluene, the product of direct nitrification of toluene (which according to many investigators is a mixture of symmetrical [2 : 3] and ortho-nitrotoluene [1 : 3]), a mixture of two azotoluenes was obtained, of which one melts at 137° and the other at 55°. The latter crystallises in short, red prisms, which were scattered about amongst the long, orange needles of the former. Petrieff's and Schultz's method consists in the reduction of orthonitrotoluene with zinc-dust. Orthonitrotoluene was purified by repeated distillation, in order to get rid of the symmetrical modification, and the product boiling at 122—123°, the purity of which was confirmed by oxidation and by reduction, was dissolved in alcohol and treated with zinc-dust in the presence of aqueous soda. After adding water, an oil was obtained, which the following day became converted into thick, red needles, melting at 55°. According to Tutkoffsky, the crystals are rhombic; axial ratio $a : b : c = 0{\cdot}875588 : 1 : 1{\cdot}013960$; observed faces ∞P and $\bar{P}\infty$. They are ruby-red and pleochroic. By reduction with sodium-amalgam in alcoholic solution, this yielded colourless crystals of the hydrazocompound (m. p. 161°), which on oxidation again gave the red

needles of orthoazotoluene (m. p. 55°). From this and other reactions, it follows that Petrieff's so-called "azoxytoluene" is this orthoazotoluene. Using Hoogewerff and van Dorp's method, the author subjected orthotoluidine to the action of potassium permanganate in alkaline solution, and the same azotoluene, melting at 55°, was obtained. Perkin's method, namely, distillation of nitrotoluene in alcoholic solution with sodium hydroxide, gives the same azotoluene (m. p. 55°), but the method cannot be recommended. From the results of these experiments it follows that orthoazotoluene melts at 55°; 100 parts of alcohol at 14·5° dissolve 6·027 parts; 100 parts of ether at 16·5° dissolve 147·66 parts. It dissolves in sulphuric acid, and is separated again on addition of water; the same with the acetic acid solution. It absorbs benzene vapour, and deliquesces to a red liquid.

B. B.

Diazo-compounds. By P. Griess (*Ber.*, **21**, 1559—1566).—*Paramidotriazobenzene*, $NH_2 \cdot C_6H_4 \cdot N<\overset{N}{N}\!\!>$, is prepared from paraphenyleneamido-oxamic acid (Abstr., 1885, 1220) in a manner exactly similar to the meta-derivative (*ibid.*, 788); it crystallises in long plates, readily soluble in the usual solvents, except water. It melts at 65°, and detonates at a higher temperature. When exposed to air it becomes reddish; it is completely decomposed by prolonged boiling with water. The *hydrochloride* crystallises in needles or plates, generally reddish; very readily soluble in cold water. The *platinochloride* forms bright yellow needles, very sparingly soluble in cold water.

Paradiazotriazobenzene was obtained by diazotising the amido-compound above described, but was not isolated. The *platinochloride*, $(N_2Cl \cdot C_6H_4 \cdot N_3)_2, PtCl_4$, crystallises in long, narrow plates, which explode violently when heated. The *perbromide*, $N_3 \cdot C_6H_4 \cdot N_2, Br_3$, is formed when a mixture of bromic and hydrobromic acid is added to an aqueous solution of the hydrobromide; it separates as a red oil, which gradually crystallises.

Paraditriazobenzene (*parahexazobenzene*), $C_6H_4\left(N<\overset{N}{N}\!\!>\right)_2$ [1 : 4], is formed when paradiazotriazobenzene perbromide is added to aqueous ammonia. The whole is distilled, and the solid distillate crystallised from ether. It crystallises in colourless, well-formed plates, which melt at 83°, and become brown when exposed to light; it is very readily soluble in ether and chloroform, sparingly soluble in alcohol, and almost insoluble in water. It has a sweet taste. When heated above its melting point, it explodes with extreme violence. It is indifferent to bases and acids in the cold, but is decomposed by prolonged boiling with strong hydrochloric acid. The compound was not analysed, but the formula assigned to it is supported by the mode of formation of the substance, as well as by analyses of the analogously constituted hexazobenzoic acid described below. Hexazobenzene can also be prepared from paratetrazobenzene.

Metamidotriazobenzoic acid, $COOH \cdot C_6H_3(NH_2)N_3$ [= 1 : 3 : 5], is prepared by boiling 1 : 3 : 5 diamidobenzoic acid dissolved in hydrochloric acid with twice the amount of oxalic acid. The *amido-*

oxalamidobenzoic acid, $COOH{\cdot}C_6H_3(NH_2){\cdot}NH{\cdot}C_2O_2{\cdot}OH$ (which forms white grains, almost insoluble in water), thus obtained is diazotised and the diazo-compound converted into the perbromide and treated with ammonia. On adding hydrochloric acid to the product, *metatriazo-oxalamidobenzoic acid*, $COOH{\cdot}C_6H_3(N_3){\cdot}NH{\cdot}C_2O_2{\cdot}OH$, is liberated. This is a yellowish substance, rather readily soluble in hot water; when boiled with dilute aqueous potash, it is decomposed into metamidotriazobenzoic and oxalic acids. Metamidotriazobenzoic acid crystallises from water in slightly reddish needles, readily soluble in cold alcohol and ether, almost insoluble in cold water. It has a slightly acid taste, and detonates when heated in the dry state. The *hydrochloride* crystallises in small needles, very readily soluble in cold water; the *platinochloride* is also described.

Metadiazotriazobenzoic acid seems not to exist in the free state. The *nitrate*, $COOH{\cdot}C_6H_3(N_3){\cdot}N_2{\cdot}NO_2$ [1 : 3 : 5], crystallises in well-formed, sparingly soluble prisms; the *perbromide*, $C_7H_4N_6Br_3$, and *platinochloride*, are also described.

Metadiazotriazoamidobenzene, $COOH{\cdot}C_6H_3(N_3)_2{\cdot}N_2{\cdot}NHPh$, is formed when metadiazotriazobenzoic acid nitrate is treated with aniline. It is very readily soluble in ether, insoluble in water, and has an intense yellow colour.

Metaditriazobenzoic acid (*hexabenzoic acid*), $COOH{\cdot}C_6H_3(N_3)_2$ [1:3:5], is prepared by dissolving metadiazotriazobenzoic acid perbromide in aqueous ammonia, and is purified by crystallisation from hot dilute alcohol (using animal charcoal), from which it separates in small, white needles, almost insoluble in boiling water, very readily soluble in ether. It has a bitter taste. When heated it explodes, with formation of a black cloud. It yields very stable salts. The *barium salt* forms almost insoluble, white needles.

Benzeneazodiphenyldisulphonic acid, $SO_3H{\cdot}C_6H_4{\cdot}N_2{\cdot}C_6H_4{\cdot}C_6H_4{\cdot}SO_3H$, is obtained by dissolving benzeneazodiphenyl (1 part) (compare *Ber.*, **21**, 911) in fuming sulphuric acid (3 to 4 parts), and heating the whole on a water-bath, until a sample is quite soluble in water. It is poured into water (6 parts), the yellowish-red crystals washed with hydrochloric acid and dried. The pure acid is prepared from the barium salt. It is readily soluble in alcohol and in cold water, and is precipitated from the latter by addition of hydrochloric acid. The *potassium salt* (with $1\frac{1}{2}$ mol. H_2O) crystallises in well-formed prisms, readily soluble in hot water; the *barium salt* forms a granulo-crystalline precipitate. The disulphonic acid is reduced by tin and hydrochloric acid, and by ammonium sulphide, with formation of a very sparingly soluble amido-acid, crystallising in narrow plates of a silvery lustre.

N. H. M.

Mixed Azo-compounds. By C. Beyer and L. Claisen (*Ber.*, **21**, 1697—1705).—*Phenylazoacetoacetaldehyde*, $COMe{\cdot}CH(CHO){\cdot}N_2Ph$, is obtained by gradually adding a dilute solution of diazobenzene chloride to an aqueous solution of sodacetoacetaldehyde. It crystallises from boiling alcohol in thick, dark-red, or thin, lighter coloured prisms, according to the concentration of the solution. It melts at 118°, and gives a crystalline copper compound with copper

acetate. The *hydrazone*, $COMe{\cdot}CH(N_2Ph){\cdot}CH{:}N{\cdot}NHPh$, prepared by mixing an alcoholic solution with phenylhydrazine, is very sparingly soluble in alcohol, from which it crystallises in yellowish-red needles melting at 218°. When boiled with glacial acetic acid, it yields a compound, $N_2Ph{\cdot}C\begin{<}{-CH{:}N-}\\{CMe{\cdot}NPh}\end{>}$, which crystallises from methyl alcohol in needles melting at 112°, and insoluble in alkalis.

An alkaline solution of phenylazoacetoacetaldehyde gives with an alkaline solution of phenylhydrazineparasulphonic acid a yellow, crystalline precipitate, probably sodium *phenylazoacetoacetaldehydehydrazideparasulphonate*, which in a sulphuric acid bath, shows the same shade as, although in a considerably less degree than, tartrazin.

Phenylazoacetylacetone, $COMe{\cdot}CH(COMe){\cdot}N_2Ph$, prepared by mixing aqueous solutions of sodium acetylacetone and diazobenzene chloride, crystallises from boiling alcohol in long, yellow needles melting at 90°.

Phenylazophenyldimethylpyrazole, $N_2Ph{\cdot}C\begin{<}{-CMe{:}N-}\\{CMe{\cdot}NPh}\end{>}$, is obtained by heating the preceding compound with phenylhydrazine at 130—140°. It crystallises from slightly acid alcohol in yellow needles, which melt at 63° and are insoluble in alkalis.

Phenylazodibenzoylmethane, $COPh{\cdot}CH(COPh){\cdot}N_2Ph$, is prepared by precipitating an aqueous alcoholic solution of sodium dibenzoylmethane with a dilute solution of diazobenzene chloride. It crystallises from boiling alcohol in yellowish-red prisms melting at 153—154°.

Phenylazotriphenylpyrazole, $N_2Ph{\cdot}C\begin{<}{-CPh{:}N-}\\{CPh{\cdot}NPh}\end{>}$, obtained by heating the preceding compound with phenylhydrazine, crystallises from boiling alcohol in yellowish-red prisms, melts at 156—157°, and is insoluble in alkalis. It is more readily soluble in light petroleum and ethyl acetate than phenylazodibenzoylmethane.

Sodium phenylazodibenzoylmethaneparasulphonate,

$$COPh{\cdot}CH(COPh){\cdot}N_2{\cdot}C_6H_4{\cdot}SO_3Na,$$

is formed by the action of diazobenzenesulphonic acid on an alkaline solution of dibenzoylmethane. It crystallises from hot water in golden-yellow leaves, and when treated with mineral acids yields the free sulphonic acid in the form of a yellow, pasty mass of microscopic needles, which colour wool greenish in a sulphuric acid bath.

The above compounds are true phenylazo-derivatives, no intramolecular change, by which they would be converted into hydrazine-derivatives, having taken place. The phenylazo-derivatives are all soluble in alkali, but are precipitated from the solution by carbonic anhydride.

The following compounds are chemically and physically similar to those already described, and are also, probably, true phenylazo-derivatives.

Phenylazobenzoylaldehyde, $COPh{\cdot}CH(CHO){\cdot}N_2Ph$, crystallises from alcohol in yellow or dark-red prisms melting at 103°.

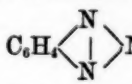
Phenylazobenzoylacetone, $COPh{\cdot}CH(COMe){\cdot}N_2Ph$, crystallises from alcohol in yellowish-red prisms melting at 99°.

Ethyl phenylazoacetylpyruvate, $COMe\cdot CH(CO\cdot COOEt)\cdot N_2Ph$, crystallises from alcohol in yellow prisms melting at 115—116°.

Ethyl phenylazobenzoylpyruvate, $COPh\cdot CH(CO\cdot COOEt)\cdot N_2Ph$, crystallises from alcohol in yellowish-red prisms melting at 116—117°.

F. S. K.

Two Bye-products in the Technical Preparation of Amidoazobenzene. By L. Gattermann and G. Wichmann (*Ber.*, **21**, 1633—1637).—The aniline recovered from the acid solution obtained in the preparation of amidoazobenzene contains amidodiphenyl sulphate and a new compound of the formula $C_{12}H_9N_3$. The latter is obtained by extracting the crude product with glacial acetic acid; on adding water to the acetic acid solution, the compound separates in lustrous needles. Its molecular weight was determined by Raoult's method. It dissolves readily in the usual solvents, melts at 109°, and boils without decomposition. It is not dissolved by acids or alkalis, and does not react with acid chlorides and with nitrous acid; it can be distilled over zinc-dust without change. When reduced with sodium in alcoholic solution, aniline and orthophenylenediamine are formed. The compound is therefore an azimide of the formula



The *nitro-derivative*, $C_{12}H_8N_4O_2$, prepared by slightly warming 2 grams of the substance with 10 c.c. of strong sulphuric acid, crystallises from glacial acetic acid in white needles melting at 275°.

The presence of amidodiphenyl sulphate is accounted for by the fact that the crude hydrochloric acid in which the aniline is dissolved, contains sulphuric acid.

N. H. M.

Phenylhydrazines. By C. Willgerodt and M. Ferko (*J. pr. Chem.* [2], **37**, 345—358).—*Picrylhydrazine* is best prepared by carefully mixing picryl chloride (1 mol.) and phenylhydrazine (2 mols.) in cold alcoholic solution. It forms pale-red scales melting at 172°. If the solution is warmed, decomposition takes place, and the melting point of the product is considerably raised. On boiling picrylhydrazine with acetic acid, strong hydrochloric acid, or dilute sulphuric acid for a short time, *dinitrosonitroazobenzene*, $Ph\cdot N_2\cdot C_6H_2(NO)_2\cdot NO_2$, crystallising in golden-yellow scales melting at 247·5° is formed, together with a substance of a brownish colour, melting at 145°, not yet investigated. On boiling with alcohol, picrylphenylhydrazine yields what is probably a mixture of trinitrohydrazobenzene, nitrosodinitrohydrazobenzene, and nitrosodinitroazobenzene.

Dinitrosonitroazobenzenesulphonic acid is formed by heating a mixture of dinitrosonitroazobenzene (5 grams) with equal parts (15 grams) of ordinary and fuming sulphuric acid at 220—230°. The acid crystallises from water in yellow needles, and from acetic acid in yellow scales, which do not melt at 360°.

α-Dinitrohydrazobenzene, $N_2H_2Ph\cdot C_6H_3(NO_2)_2$, is best prepared by digesting finely-powdered α-dinitrochlorobenzene (5 grams) with phenylhydrazine (5·5 grams) in alcoholic solution for eight days. It

crystallises from hot alcohol in beautiful red scales melting at 120°. When shaken with mercuric oxide, it yields α-dinitroazobenzene, which crystallises from alcohol in small needles melting at 116—117° (uncorr.).

Dinitrosoazobenzene may be prepared by heating α-dinitrochlorobenzene with phenylhydrazine and alcohol in sealed tubes at 120—130°. It crystallises in needles of a brownish colour melting at 175°.

Orthonitrometachlorhydrazobenzene is formed when Laubenheimer's dinitrochlorobenzene (3 grams) is heated with phenylhydrazine (3·3 grams) and alcohol. It crystallises in short, red prisms melting at 139—140° (uncorr.).

Orthonitrometachlorazobenzene, $Ph{\cdot}N_2{\cdot}C_6H_3{\cdot}NO_2Cl$ [N : NO_2 : Cl = 1 : 2 : 5], is prepared by treating the preceding hydrazo-compound with mercuric oxide. It forms small, red needles melting at 94°.

Orthonitrosometachlorazobenzene is formed by boiling the corresponding hydrazo-compound with water or acids. It melts at 136—137.

The authors have failed in their attempts to obtain an unsymmetrical picrylhydrazine by heating together picryl chloride (8·6 grams) and phenylhydrazine (5 grams) in alcoholic solution. G. T. M.

New Method for the Preparation of Biguanides. Derivatives of Phenylbiguanide. By A. Smolka and A. Friedreich (*Monatsh.*, **9**, 227—241).—The hydrochlorides of biguanide, ethylbiguanide, and phenylbiguanide can be prepared by heating an alcoholic solution of dicyandiamide with ammonium chloride, ethylamine hydrochloride, and aniline hydrochloride respectively.

Copper phenylbiguanide, $(C_8H_{10}N_5)_2Cu + 1\frac{1}{2}H_2O$, is obtained by treating the hydrochloride (see below) with warm soda. It is a dark-rose coloured, microscopic, crystalline powder, which decomposes and turns brown when boiled with water, or, more quickly, when heated alone at 130—135°. It is very sparingly soluble in cold, more readily soluble in hot water; the aqueous solution absorbs carbonic anhydride from the air and becomes turbid owing to the separation of the carbonate. It has strongly basic properties, liberates ammonia from its salts, precipitates metallic hydroxides from solutions of metallic salts, and combines with acids. The *hydrochloride*, $(C_8H_{10}N_5)_2Cu,2HCl + 1\frac{1}{2}H_2O$, prepared by digesting phenylbiguanide hydrochloride with freshly-precipitated copper hydroxide, crystallises in carmine-red aggregates which slowly decompose when heated above 140°. It is readily soluble in water, but is decomposed with separation of a brown product, when the solution is boiled for some time. Alkalis give rose-coloured precipitates of the copper base when added to the aqueous solution and soluble sulphates, carbonates, phosphates, chromates, and oxalates precipitate the corresponding salt of copper phenylbiguanide. The *sulphate*, $(C_8H_{10}N_5)_2Cu,H_2SO_4,1\frac{1}{2}H_2O$, forms rose-coloured, microscopic needles which decompose when heated at about 150°. It is not decomposed when boiled with water, in which it is very sparingly soluble. The *chromate*, $(C_8H_{10}N_5)_2Cu,H_2CrO_4,H_2O$, is a yellowish-green, microscopic, granular powder, which decomposes when heated at about 150°. It is more sparingly soluble in water than the sulphate, and does not decompose when boiled with water.

Nickel phenylbiguanide, $(C_8H_{10}N_5)_2Ni$, prepared by precipitating the hydrochloride with soda, is a yellow powder which is almost totally insoluble in water, and is not decomposed either when heated at 150° or when boiled with water. The *hydrochloride*, $(C_8H_{10}N_5)_2Ni,2HCl$, prepared in like manner to the copper salt, forms yellow, granular crystals which are readily soluble in water and decompose when heated to about 150°. The aqueous solution is yellow, decomposes when heated, and behaves towards reagents similarly to the corresponding copper salt. The *sulphate*, $(C_8H_{10}N_5)_2Ni,H_2SO_4 + 1\frac{1}{2}H_2O$, crystallises in orange-yellow, microscopic plates which decompose when heated at about 150°. It is very sparingly soluble in water, and is not decomposed when boiled with it.

Cobalt phenylbiguanide, $(C_8H_{10}N_5)_2Co + 1\frac{1}{2}H_2O$, is a dark-rose coloured, microscopic, granular powder, which is far more readily soluble in water than the copper or nickel base. It forms large, raspberry-red leaves when a dilute aqueous solution is evaporated slowly; it has strongly basic properties. It is decomposed when heated at about 145° or when boiled with water. The *hydrochloride*, $(C_8H_{10}N_5)_2Co,2HCl + 1\frac{1}{2}H_2O$, forms small, raspberry-red plates which are tolerably easily soluble in water, forming a wine-red coloured solution. It is decomposed when boiled with water or when heated at about 150°, and it behaves towards reagents like the corresponding copper and nickel salts. The *sulphate*, $(C_8H_{10}N_5)_2Co,H_2SO_4 + H_2O$, forms silky, carmine-red needles which are sparingly soluble in water, and decompose when heated to about 150°. It is not decomposed when boiled with water. F. S. K.

Safranines and related Dyes. By R. Nietzki and R. Otto (*Ber.*, **21**, 1590—1598; compare Abstr., 1887, 250).—When an alcoholic solution of phenosafranine is treated with sulphuric acid just sufficient to produce a violet coloration, and boiled with an excess of sodium nitrite, a magenta-coloured solution is obtained which is not fluorescent. The new compound thus obtained is less strongly basic than safranine, and can be liberated from its salts by caustic alkalis, but not by carbonates. It is purified by precipitation with zinc chloride and hydrochloric acid. The *nitrate*, $C_{18}H_{13}N_3,HNO_3$, *sulphate*, $C_{18}H_{13}N_3,H_2SO_4$, and *platinochloride* were prepared. The *acetyl*-derivative is violet, and yields yellow, sparingly soluble salts. The compound $C_{18}H_{13}N_3$, in the form of its salts, dissolves in water with red colour, and dyes wool and silk red, but with a much more bluish shade than safranine does. Hydrochloric acid dissolves it with green coloration which changes at once to red on addition of water.

The *phenosafranine base*, $C_{18}H_{16}N_4O$, is prepared by treating the sulphate with the exact amount of baryta. On evaporating the solution with exclusion of air, plates of a golden lustre separate: it is best to evaporate in a vacuum at 40°. It is readily soluble in alcohol, sparingly soluble in water, and almost insoluble in ether. Water decomposes it with formation of the base $C_{18}H_{14}N_4$.

Safranol, $C_{18}H_{19}N_2(OH)_2$, is obtained by boiling phenosafranine hydrochloride with an excess of alcoholic potash for three days; the alcohol is evaporated, water and then an excess of acetic acid

added; the brown crystals thus obtained are dissolved in ammonia and precipitated with hydrochloric acid. It separates in large, brass-coloured needles, is almost insoluble in water, alcohol, and glacial acetic acid, but readily soluble in caustic alkali and ammonia, with carmine-red colour. Strong sulphuric acid dissolves it with brown colour. The *diacetyl*-derivative crystallises in plates of a metallic lustre, insoluble in ammonia. When phenosafranine is boiled for several days with zinc-dust and hydrochloric acid, the *compound* $C_{18}H_{19}N_3O$ is formed. This crystallises from water in long, colourless needles, readily soluble in alcohol, less soluble in ether, benzene, and hot water, and melts at 130°, losing water. No crystalline salts could be obtained. The *acetyl*-derivative, $C_{18}H_{18}N_3OAc$, forms broad needles melting at 173°, readily soluble in acids, and yields a yellow crystalline *platinochloride*, $(C_{18}H_{18}N_3OAc)_2,H_2PtCl_6$. When a solution of the base in an excess of dilute sulphuric acid is treated with sodium nitrite and then with alcohol, a new *base*, of the formula $C_{18}H_{18}N_2O$, is obtained. The latter crystallises from dilute alcohol in broad needles melting at 117°; it yields no acetyl-derivative, and cannot be diazotised. The *platinochloride*, $(C_{18}H_{18}N_2O)_2,H_2PtCl_6$, is sparingly soluble.

The behaviour of the base, $C_{18}H_{19}N_3O$, when heated makes it probable that the oxygen in it is present in the form of water, and that the compound, as well as the compound $C_{18}H_{18}N_2O$, are the respective hydrates of the bases $C_{18}H_{17}N_3$ and $C_{18}H_{16}N_2$. N. H. M.

Chemical Process which takes place in the Dyeing of Wool and Silk with Basic Coal-tar Dyes. By E. KNECHT (*Ber.*, **21**, 1556—1558).—In order to determine whether the taking up of dyes by fibres is a mechanical or chemical process, quantitative experiments were made in which wool and silk respectively were boiled with solutions of weighed amounts of rosaniline hydrochloride, diamido-azobenzene hydrochloride, and methyl violet. 0·2 gram of rosaniline hydrochloride was dissolved in 500 c.c. of water and boiled with wool until the solution was colourless. The liquid was then neutral and contained ammonia. A hydrochloric acid determination showed that the whole of the hydrochloric acid originally in the magenta remained in the decolorised solution. Similar results were obtained from the other experiments. The taking up of dyes by animal fibres involves, therefore, a quantitative chemical change, and is not a mechanical process merely. It is probable that very complex compounds are formed. N. H. M.

Derivatives of Diphenylphosphorous Chloride and Diphenylphosphine. By C. DÖRKEN (*Ber.*, **21**, 1505—1515).—*Diphenylbenzylphosphine chloride*, $C_7H_7{\cdot}PPh_2Cl_2$, obtained by heating diphenylphosphorous chloride at 180° with excess of benzyl chloride, forms slightly yellow prisms, melts at 187°, and is insoluble in ether and benzene. When treated with alcohol or water, it is quickly converted into diphenylbenzylphosphine oxide, melting at 195—196°.

Diphenyldimethylphosphonium iodide, PMe_2Ph_2I, is prepared by heating diphenylphosphorous chloride with methyl iodide.

Trinitrodiphenylbenzylphosphine oxide, $NO_2{\cdot}C_7H_6{\cdot}P(C_6H_4{\cdot}NO_2)_2$, formed by treating the oxide with a mixture of fuming nitric acid and concentrated sulphuric acid, separates from glacial acetic acid in colourless crystals which melt at 206°, are insoluble in alcohol, ether, &c., but moderately soluble in glacial acetic acid. Diphenylphosphine can be obtained by heating diphenylphosphorous chloride at 230° with zinc. It is a colourless, oily liquid of disagreeable odour, insoluble in water, but readily soluble in ether, alcohol, and benzene. On exposure to the air, it is oxidised to diphenylphosphinic acid. It combines with methyl iodide, acetic chloride, and benzoic chloride.

Diphenylphosphonium chloride, PH_2Ph_2Cl, obtained in the form of a white, crystalline mass by treating diphenylphosphorous chloride with hydrogen chloride, is sparingly soluble in ether and quickly decomposes on exposure to the air. The *platinochloride*, $(PH_2Ph_2Cl)_2,PtCl_4$, is a yellow powder.

Diphenylphosphonium iodide, formed by the combination of diphenylphosphine with concentrated hydriodic acid, is a yellowish-white, unstable compound.

Tetraphenyldiphosphine, $PPh_2{\cdot}PPh_2$, obtained by warming diphenylphosphorous chloride with diphenylphosphine, melts at 67°, boils at about 400°, and gradually oxidises on exposure to the air. It is converted into diphenylphosphinic acid when warmed with nitric acid. Diphenylphosphine and carbon bisulphide combine when heated together, to form a compound $PPh_2{\cdot}CS{\cdot}S{\cdot}PH_2Ph_2$, which crystallises from warm alcohol in shining forms melting at 157°. It is readily soluble in carbon bisulphide, glacial acetic acid, and warm alcohol, but insoluble in ether.

Paradiphenyltolylphosphine, $PPh_2{\cdot}C_6H_4Me$, prepared by the action of sodium on a mixture of diphenylphosphorous chloride and para-bromotoluene in ethereal solution, crystallises from ether or alcohol in colourless prisms melting at 68°. It is a feeble base, dissolves readily in concentrated hydrochloric acid, but is reprecipitated from the solution by water.

Diphenyltolylphosphine oxide, $POPh_2{\cdot}C_6H_4Me$, is obtained by treating the phosphine first with bromine and then with alkali. It crystallises from alcohol, melts at 129—130°, and is only sparingly soluble in ether.

Diphenyltolylphosphine sulphide, $PSPh_2{\cdot}C_6H_4Me$, is formed when a carbon bisulphide solution of the phosphine and sulphur is evaporated. It crystallises from alcohol in long, silky needles, which melt at 139°, are readily soluble in carbon bisulphide, moderately soluble in alcohol, but insoluble in ether.

Ditolylphenylphosphine, $PPh(C_6H_4Me)_2$, obtained by the action of sodium on an ethereal solution of phosphenyl chloride and para-bromotoluene, crystallises from alcoholic ether in small, colourless needles, melts at 57°, and is readily soluble in ether, but only sparingly in alcohol.

Dinitrodiphenylphosphinic acid, $(NO_2{\cdot}C_6H_4)_2PO{\cdot}OH$, is prepared by treating diphenylphosphinic acid with a mixture of concentrated

sulphuric acid and fuming nitric acid. It melts at 268°, and is only sparingly soluble in alcohol, ether, benzene, and glacial acetic acid. When heated in a test-tube, it explodes. The *ammonium* salt, $(C_6H_4\cdot NO_2)_2PO\cdot ONH_4$, crystallises in small, yellow columns which melt at 260°, and are sparingly soluble in alcohol and cold water. The *potassium* salt, $(C_6H_4NO_2)_2PO\cdot OK + 2H_2O$, is a yellow, crystalline powder, which is readily soluble in water, but more sparingly soluble in alcohol. The *barium*, *lead*, and *silver* salts are yellowish, crystalline compounds.

Diamidodiphenylphosphinic acid, obtained by reducing the nitro-compounds, forms small, brown crystals, melts at 276° with decomposition, and is only sparingly soluble in almost all solvents.

F. S. K.

Derivatives of Dimethylaniline containing Phosphorus; and Mercury Dimethylaniline. By A. Scheuk and A. Michaelis (*Ber.*, **21**, 1497—1504).—*Dimethylamidophenylphosphorous chloride*, $NMe_2\cdot C_6H_4\cdot PCl_2$ [$PCl_2 : NMe_2 = 1 : 4$], is obtained by mixing phosphorus trichloride with dimethylaniline and gradually adding aluminium chloride. It forms a slightly yellow, deliquescent, crystalline mass, melts at 66°, and boils at 250° (120 mm. pressure) with partial decomposition. It is sparingly soluble in light petroleum, more readily soluble in ether, and readily soluble in benzene and phosphorus trichloride. It dissolves in water with evolution of heat and a strong phosphine smell. A white powder, probably the *hydrochloride*, $PCl_2\cdot C_6H_4\cdot NMe_2,HCl$, soon separates from solutions of this compound owing to absorption of moisture.

Sodium dimethylamidophenylphosphinite, $NMe_2\cdot C_6H_4\cdot PO_2HNa + 2H_2O$, obtained by evaporating an aqueous solution of the preceding compound and treating the residue with soda, forms large well-defined crystals.

Dimethylamidophenylphosphinous acid, $NMe_2\cdot C_6H_4\cdot PO_2H_2$, prepared by decomposing the lead salt with hydrogen sulphide, crystallises in white needles which melt at 162°, are readily soluble in water and hot alcohol, but only sparingly in cold alcohol. The acid is decomposed into dimethylaniline and phosphorous acid when heated in aqueous or dilute hydrochloric acid solution, but it is very stable in alkaline or alcoholic solution. Dimethylaniline, phosphorus and hydrogen phosphide are obtained when the acid is heated alone.

Dimethylamidophenylphosphonic acid, $NMe_2\cdot C_6H_4\cdot PO(OH)_2$, is prepared by oxidising sodium dimethylamidophenylphosphinite in alcoholic solution with mercuric chloride. From alcohol, it separates in crystals which melt at 133°, and are readily soluble in water and alcohol. In aqueous solution this acid seems to be as unstable as dimethylamidophenylphosphinous acid.

Paramercurodimethylaniline, $Hg(C_6H_4\cdot NMe_2)_2$, is formed when parabromodimethylaniline is dissolved in coal-tar xylene, ethyl acetate added, and the mixture boiled with sodium amalgam. It crystallises from benzene and alcoholic benzene with 1 mol. of benzene in slightly yellow needles which quickly become dull and crumble to a white powder on exposure to the air, owing to the volatilisation of the benzene. It melts at 169°, is sparingly soluble in alcohol and ether,

but dissolves readily in benzene and chloroform. Soda precipitates the base unchanged from its solution in dilute hydrochloric acid, but when heated with hydrochloric acid it is decomposed. When treated with phosphorus trichloride, it yields dimethylamidophenylphosphorous chloride.

Dimethylamidotriphenylphosphine, $NMe_2 \cdot C_6H_4 \cdot PPh_2$, is formed by the action of sodium on a benzene solution of dimethylamidophenylphosphorous chloride and chlorobenzene. It crystallises from hot alcoholic ether in forms which melt at 152°, are readily soluble in benzene, but tolerably sparingly soluble in alcohol and ether. This compound is only a feeble base, and is precipitated from its solution in concentrated hydrochloric acid by water. It combines readily with benzyl chloride and alkyl iodides, and can be readily converted into the corresponding oxide and sulphide.

Hexamethyltriamidotriphenylphosphine, $P(C_6H_4 \cdot NMe_2)_3$ (Hanimann, this Journal, 1877, ii, 417), is obtained as a bye-product in the preparation of dimethylamidophenylphosphorous chloride. It forms slender, colourless needles, melts at 273°, and turns blue on exposure to the air. It is a very stable compound, is sparingly soluble in cold, more readily in hot alcohol, and is readily soluble in chloroform and dilute hydrochloric acid.

A white powder, $C_{12}H_{10}NPO$, is formed when diphenylamine and phosphorus trichloride are heated with zinc chloride in sealed tubes. This compound yields nitro- and amido-derivatives which are, however, very sparingly soluble and mostly amorphous.

F. S. K.

Xylylphosphorus Compounds and Toluphosphonic Acids. By J. WELLER (*Ber.*, **21**, 1492—1497; compare Abstr., 1887, 824).—*Lead α-toluphosphonate*, $COOH \cdot C_6H_3Me \cdot PO:O_2:Pb + H_2O$, crystallises from dilute acetic acid in transparent needles which are insoluble in water. *Silver α-toluphosphonate* and *silver β-toluphosphonate*,

$$COOAg \cdot C_6H_3Me \cdot PO(OAg)_2,$$

are white, amorphous compounds which are sparingly soluble in water, but readily soluble in ammonia and nitric acid.

β-Toluphosphonic acid chloride, $COCl \cdot C_6H_3Me \cdot POCl_2$, formed by the action of phosphorus pentachloride on the acid, boils at 249° (147 mm. pressure), and is readily decomposed by water.

Paraxylylphosphorous chloride, $C_6H_3Me_2 \cdot PCl_2$, prepared by acting on a mixture of phosphorous chloride and paraxylene with aluminium chloride, is a colourless, strongly refractive liquid which boils at 253—254°, and solidifies at −30° to colourless needles.

Paraxylylphosphorous tetrachloride, $C_6H_3Me_2 \cdot PCl_4$, produced when the preceding compound is treated with chlorine, forms a colourless or slightly yellow mass which melts at 60°.

Paraxylylphosphorous oxychloride, $C_6H_3Me_2 \cdot POCl_2$, is formed by treating the tetrachloride with water or sulphurous anhydride. It is a thick, colourless liquid which boils at 280—281°.

Paraxylylphosphinous acid, $C_6H_3Me_2 \cdot PO_2H_2$, is formed when the corresponding chloride is treated with warm water.

Paraxylphosphonic acid, $C_6H_3Me_2 \cdot PO(OH)_2$, obtained by decomposing paraxylyltetra- or oxy-chloride with water, crystallises in colourless needles and melts at 179—180°. It is moderately soluble in water, dissolves readily in alcohol, but is only sparingly soluble in ether. When heated, it is decomposed, yielding metaphosphoric acid and paraxylene. The acid *potassium* salt, $C_6H_3Me_2 \cdot PO_3HK$, and the *barium* salt, $C_6H_3Me_2 \cdot PO_3Ba$, are crystalline.

Paranitroxylylphosphonic acid, $NO_2 \cdot C_6H_2Me_2 \cdot PO(OH)_2$, is prepared by nitrating the acid. It crystallises in needles, melts at 224°, and is readily soluble in alcohol, but only sparingly in ether.

Paratoluphosphonic acid, $COOH \cdot C_6H_3Me \cdot PO(OH)_2$, is formed when paraxylylphosphonic acid is oxidised with potassium permanganate in alkaline solution. It crystallises from dilute alcohol in colourless or slightly yellow, slender needles melting at 278°. It is readily soluble in alcohol, sparingly soluble in water, and almost insoluble in ether. When heated, it yields paratoluic acid and metaphosphoric acid with partial decomposition.

Paratoluphosphonic chloride, $COCl \cdot C_6H_3Me \cdot POCl_2$, forms a colourless crystalline mass which melts at 62°, and is readily decomposed by water. F. S. K.

Action of Methylamine and Ethylamine on Salicaldehyde. By M. Dennstedt and J. Zimmermann (*Ber.*, **21**, 1553—1554).—When a slow stream of methylamine (1 mol.) is passed through salicaldehyde, yellow crystals of the additive compound (corresponding with aldehyde-ammonia) separate. Subsequently the liquid becomes warm, the crystals redissolve, and a separation of water takes place. The product of the reaction is extracted with ether and dried with calcium chloride; on distilling off the ether, a yellow oil is obtained of the composition represented by the formula C_8H_9NO. It boils at 229°. When treated with zinc chloride, it yields only a resin.

In a similar manner, ethylamine reacts with salicaldehyde with formation of the *compound* $C_9H_{11}NO$. This is a heavy, yellow oil which is soluble in water and boils at 237°. N. H. M.

Tetrachlorobenzoic Acid from Tetrachlorophthalic Acid. By P. Tust (*Ber.*, **21**, 1532—1534; compare Abstr., 1887, 1046).—Barium tetrachlorobenzoate, $(C_6HCl_4 \cdot COO)_2Ba + 3\frac{1}{2}H_2O$, is moderately soluble in hot water, from which it crystallises in colourless, concentric needles which crumble on exposure to the air.

Tetrachloraniline, $C_6HCl_4 \cdot NH_2$ [$Cl_4 = 2 : 3 : 4 : 5$], is obtained when tetrachloranthranilic acid is heated. The last-named compound is prepared by nitrating tetrachlorobenzene and reducing the nitro-derivative obtained. The product is saturated with alkali and distilled with steam, when tetrachloraniline separates as a white, crystalline mass. It is easily soluble in alcohol, ether, benzene, and glacial acetic acid, but more sparingly in light petroleum, from which it crystallises in concentrically grouped needles melting at 118°.

Tetrachloracetanilide, $C_6HCl_4 \cdot NHAc$, crystallises from glacial acetic

acid in colourless, shining needles, melts at 154°, and is readily soluble in alcohol, ether, and glacial acetic acid. F. S. K.

Metahomo-anthranilic Acid and its Derivatives. By S. NIEMENTOWSKI (*Ber.*, **21**, 1534—1541). — *Orthonitroparatolunitrile*, [CN : NO_2 : Me = 1 : 2 : 4], is obtained from the corresponding toluidine by Sandmeyer's method. It is very sparingly soluble in boiling water, from which it crystallises in long, yellow needles melting at 101°. It dissolves easily in all organic solvents, and can be sublimed or distilled with steam.

Orthamidoparatolunitrile, [CN : NH_2 : Me = 1 : 2 : 4], prepared by reducing the nitro-compound with tin and hydrochloric acid, crystallises from dilute alcohol in thin, mostly hexagonal leaves, but sometimes in short, lance-shaped forms melting at 94°. It is insoluble in cold, but soluble in hot water, and readily soluble in alcohol, chloroform, acetone, benzene, ether, and light petroleum. It yields metatoluidine, ammonia, and carbonic anhydride when heated at 180° with hydrochloric acid. The *acetyl*-derivative, $CN{\cdot}C_6H_3Me{\cdot}NHAc$, which melts at 133°, is insoluble in cold, and only sparingly soluble in hot, water. It is easily soluble in alcohol, acetone, benzene, chloroform, ether, and light petroleum, but insoluble in cold acids and alkalis.

Orthamidoparatoluylamide, $NH_2{\cdot}C_6H_3Me{\cdot}CONH_2$ [$CONH_2$: NH_2 : Me = 1 : 2 : 4], is prepared by heating orthamidoparatolunitrile with dilute potash until the whole has just dissolved. It crystallises in white, very thin leaves or silky needles, melts at 146—147°, and is readily soluble in alcohol, acetone, chloroform, boiling water, and boiling benzene, sparingly soluble in boiling light petroleum, and insoluble in carbon bisulphide. The *hydrochloride* crystallises in needles which are very readily soluble in water.

Anhydracetylorthamidoparatoluylamide (*β-methyl-δ-hydroxymetatoluquinazoline*), $C_{10}H_{10}N_2O$, is obtained when orthamidoparatoluylamide is boiled with an excess of acetic anhydride. It crystallises from boiling water in long, slender, white needles, melts at 255°, and is easily soluble in alcohol, acetone, chloroform, boiling benzene, and acids, but sparingly in ether, boiling water, and boiling light petroleum.

A similarly constituted condensation product is formed from orthamidoparatoluylamide and ethyl acetoacetate.

Toluylazimide, $C_6H_3Me{<}\overset{CO}{N{:}N}{>}NH$, is formed by the action of nitrous acid on orthamidoparatoluylamide. It crystallises in long, white needles, melts at 226° with decomposition, and is almost totally insoluble in water, sparingly soluble in benzene, but dissolves easily in alcohol and boiling chloroform. It dissolves in very dilute alkalis and in concentrated acids, but is precipitated unchanged on adding water to its solution in acids.

Metahomo-anthranilic acid, $NH_2{\cdot}C_6H_3Me{\cdot}COOH$ [$COOH$: NH_2 : Me = 1 : 2 : 4], prepared by boiling orthamidoparatolunitrile or the corresponding amide with potash until the evolution of ammonia ceases, crystallises from alcohol in needles or leaves, which melt at 177° with evolution of carbonic anhydride. It is very easily soluble in

boiling alcohol, ether, and benzene, but only sparingly in boiling water, light petroleum, and carbon bisulphide. It forms salts both with acids and bases, and is converted into metahomosalicylic acid, melting at 174°, by the action of nitrous acid. When heated with carbamide, it yields a condensation product which melts at 360°; when boiled with ethyl acetoacetate, a compound is obtained which crystallises from alcohol in long, slender needles. The *acetyl*-derivative crystallises from dilute alcohol in thick needles, melts at 183°, and is readily soluble in alcohol, chloroform, and boiling benzene, but only sparingly in water and light petroleum. *Ammonium orthacetamidoparatoluate* crystallises in needles which are very soluble in water. The *silver* salt is a white, crystalline compound which is sparingly soluble in boiling water. F. S. K.

Anhydrides of Cresotic Acid. By G. Bargioni and H. Schiff (*Annalen*, **245**, 42—44).—By the action of phosphorus oxychloride on α-cresotic acid, two anhydrides appear to be formed. The authors regard the portion soluble in alcohol as *tetracresotide*, $C_{32}H_{28}O_9$, and the insoluble portion as *hexacresotide*, $C_{48}H_{38}O_{13}$. The former melts at about 130° and the latter at about 250°. At 160°, ammonia converts hexacresotide into a mixture of ammonium creasotate and α-cresotamide, $OH{\cdot}C_6H_3Me{\cdot}CONH_2$ [Me : OH : $CONH_2$ = 1 : 4 : 5]. Aniline converts the hexacresotide into α-cresotanilide. This substance melts at 53° and forms crystalline scales. W. C. W.

Dicresoldicarboxylic Acid. By A. Deninger (*Ber.*, **21**, 1639—1641).—*Dicresoldicarboxylic acid*, $C_{16}H_{14}O_6$, is prepared by neutralising orthodicresol with alcoholic soda, drying the product at 180° in a stream of hydrogen, and heating it in an autoclave with carbonic anhydride for four hours at 160°, when the reaction takes place almost quantitatively. The product is dissolved in water and precipitated with hydrochloric acid; it is twice crystallised from pyridine, the salt then decomposed with hydrochloric acid, and the free acid crystallised from alcohol. It is insoluble in water, sparingly soluble in alcohol, ether, and chloroform, and does not melt at 290°. It gives a blue reaction with ferric chloride. The salts of the alkalis are readily soluble and amorphous; the salts of the heavy metals are insoluble in water, and are also amorphous. The *diacetyl-compound*, $C_{20}H_{18}O_8$, crystallises in needles decomposing at 163°, insoluble in water, sparingly soluble in alcohol.

Dinitrodicresol, $C_{14}H_{12}N_2O_6$ (Gerber, this vol., p. 484), is obtained by heating finely powdered dicresoldicarboxylic acid with nitric acid (sp. gr. = 1·3) until gas is evolved. It crystallises from pyridine in well-formed, reddish-yellow needles which may be washed with alcohol or ether, but which, when kept in a desiccator, gives up pyridine without losing their form; the decomposition is also brought about by water or, better, by aqueous hydrochloric acid. The free nitrophenol is soluble in toluene and in glacial acetic acid. It is very readily reduced by zinc and acetic acid to the corresponding diamide. N. H. M.

Ethyl Phenacylbenzoylacetate. By S. Kapf and C. Paal (*Ber.*, **21**, 1485—1492; compare Abstr., 1884, 598, 1117; and 1885, 248).—*Ethyl phenacylbenzoylacetate*, $COPh{\cdot}CH(COOEt){\cdot}CH_2{\cdot}COPh$, is obtained by the action of phenacyl bromide (acetophenone bromide) on ethyl sodiobenzoylacetate. It is insoluble in water, but soluble in almost all ordinary organic solvents, and crystallises from ether or light petroleum in large, transparent, well-defined forms melting at 55—56°. When treated with dilute potash at the ordinary temperature, it yields benzoic acid and benzoylpropionic acid; if a slightly impure product is hydrolysed, an insoluble residue is formed, from which a crystalline product melting at 142—145° is obtained. This compound appears to be *ethyl diphenacylbenzoylacetate*,

$$COPh{\cdot}C(COOEt)(CH_2{\cdot}COPh)_2.$$

Phenylacetylenebenzoylacetic acid, $COPh{\cdot}CH(COOH)C{\vdots}CPh$, is formed when the preceding compound is treated with alcoholic potash, or when it is distilled in a vacuum. This acid is insoluble in water, sparingly soluble in light petroleum, but dissolves tolerably readily in hot alcohol, ether, benzene, and glacial acetic acid. It crystallises in yellow needles, melts at 135°, and can be distilled in small quantities with slight decomposition. The *potassium* salt, $C_{17}H_{11}O_3K + 2H_2O$, is very sparingly soluble in cold, more readily in hot, water, and crystallises in very thin, yellow, long, flexible needles or threads. It is tolerably readily soluble in hot alcohol, but insoluble in an excess of alkali. An aqueous solution of the salt gives precipitates with the chlorides of calcium, barium, and mercury, with the sulphates of manganese and copper, and with silver nitrate.

α-α'-Diphenylfurfuran-β-carboxylic acid, $O<\begin{matrix}CPh\\CPh{\vdots}CH\end{matrix}>C{\cdot}COOH$, is produced when phenylacetylenebenzoylacetic acid is boiled with fuming hydrochloric acid and a little alcohol. It crystallises from alcohol in white needles, melts at 217°, and can be distilled with only slight decomposition. This acid is insoluble in water and light petroleum, but dissolves tolerably readily in warm ether, alcohol, glacial acetic acid, or benzene. It yields benzoic acid when oxidised with potassium permanganate in alkaline solution. The *sodium* salt, $C_{17}H_{11}O_3Na$, is readily soluble in alcohol or warm water, but is precipitated from its aqueous solution in slender needles by an excess of alkali. An aqueous solution gives precipitates with the chlorides of calcium, zinc, mercury, manganese, barium, and iron; with the sulphates of magnesium, iron, and copper, and with silver nitrate. The *ethyl* salt crystallises from ether in large forms melting at 82°.

2 : 5 *Diphenylfurfuran*, $O<\begin{matrix}CPh{\vdots}CH\\CPh{\vdots}CH\end{matrix}>$, is obtained by heating phenylacetylenebenzoylacetic acid at 150—160° for two to three hours with fuming hydrochloric acid; also by distilling phenylacetylenebenzoylacetic acid or diphenylfurfurancarboxylic acid over zinc-dust. It is insoluble in water and aqueous alkalis, but readily soluble in ether, alcohol, glacial acetic acid, benzene, and light petroleum; it crystallises from dilute alcohol in large, elongated, shining leaves or

at needles which melt at 91° and boil at 343—345° without decomposition.

Ethyl α-α'-diphenylpyrroline-β-carboxylate, $<^{NH\cdot CPh}_{CPh:CH}>C\cdot COOEt$, is prepared by acting on ethyl phenylacetylenebenzoylacetate with alcoholic ammonia at the ordinary temperature. It is sparingly soluble in hot alcohol, ether, and benzene; more readily soluble in hot glacial acetic acid, from which it crystallises in large, yellow, shining needles or prisms melting at 217°.

α-α'-Diphenylpyrroline-β-carboxylic acid, $<^{NH\cdot CPh}_{CPh:CH}>C\cdot COOH$, obtained by hydrolysing the preceding compound, is readily soluble in boiling glacial acetic acid, but tolerably sparingly soluble in hot alcohol, from which it crystallises in orange-red needles melting at 261°. It sublimes with decomposition, but is not acted on by mineral acids, so that neither the acid nor the ethyl salt shows the pinewood reaction. F. S. K.

Isomerides of Tannic Acid. By H. Schiff (*Annalen*, **245**, 35—42).—Pyrogallocarboxylic acid, prepared by the action of potassium hydrogen carbonate and water on pyrogallol, as described by Albrecht and Will (Abstr., 1884, 1335), melts with decomposition at 206—208° if slowly heated, and at 215—220° if the temperature is rapidly raised. It is converted into *dipyrogallocarboxylic acid* by the action of phosphorus oxychloride. The new acid, $C_{14}H_{10}O_9$, is a yellow powder with an astringent taste. After moistening with alcohol, it dissolves readily in water.

The aqueous solution resembles tannic acid in its reactions, but is decomposed by boiling, yielding pyrogallol. The barium salt, $Ba(C_{14}H_9O_9)_2$, is a grey powder. The acid forms a pentacetyl-derivative, $C_{14}H_5Ac_5O_9$.

Ethyl pyrogallocarboxylate has been described by Albrecht and Will (*loc. cit.*). The yield is increased by saturating the well-cooled alcoholic solution of pyrogallocarboxylic acid with hydrogen chloride under pressure.

Diphloroglucinolcarboxylic acid, prepared by the action of phosphorus oxychloride on phloroglucinolcarboxylic acid, is partly decomposed by dilute sulphuric acid at 150°, and is partly converted into an anhydride resembling phlobaphene. W. C. W.

Action of Phosphorus Pentachloride on Ethyl Succinosuccinate. By S. Levy and A. Andreocci (*Ber.*, **21**, 1463—1468).—*Dichlorodihydroterephthalic acid*, $C_6H_4Cl_2(COOH)_2$, is obtained when ethyl succinosuccinate (1 mol.) is heated with phosphorus pentachloride (4 mols.) in a reflux apparatus in a water-bath until hydrogen chloride ceases to be evolved, and, after removal of the phosphorus chlorides by distillation, is separated by repeated extraction with water at 99° from a yellow compound simultaneously formed. It crystallises from water in lustrous, white scales, becomes brown at 210°, melts at 272—275° with decomposition, and is readily soluble in alcohol, ether, and acetone, sparingly soluble in chloroform, benzene,

and carbon bisulphide. The *barium* salt with 3 mols. H_2O crystallises in small, pale-rose coloured prisms, and is sparingly soluble in hot water; the *calcium* salt with 4 mols. H_2O is almost insoluble in water; the *silver* salt forms small, asbestos-like needles, and is insoluble in water; and the *sodium hydrogen* salt with 3 mols. H_2O crystallises in colourless needles grouped in branch-like forms, and is very soluble in water. The *ethyl* salt, $C_6H_4Cl_2(COOEt)_2$, crystallises from dilute alcohol in small, lustrous prisms, melts at 70—71°, and is readily soluble in ether, benzene, and chloroform, yielding solutions which show a bluish fluorescence; the *methyl* salt, $C_6H_4Cl_2(COOMe)_2$, crystallises in thin tablets arranged in fan-like forms, and melts at 109—110°.

Paradichloroterephthalic acid, $C_6H_2Cl_2(COOH)_2$, is formed from the dihydro-acid by oxidation in aqueous solution with dilute nitric acid (20 per cent.). It crystallises from water in colourless, hair-like needles, does not melt at 300°, and can be sublimed by careful heating. Its *methyl* salt melts at 131—132°. W. P. W.

Action of Alkaline Sulphinates on Trihalogen-substituted Hydrocarbons. By R. Otto and W. Otto (*Ber.*, **21**, 1691—1697; compare this vol., pp. 482 and 483).—Chloroform and sodium benzenesulphinate do not react when heated together in sealed tubes at 160° for eight hours. Ethylenediphenylsulphone is the chief product obtained when methylchloroform is heated with sodium benzenesulphinate at 130—140°. Benzenesulphonic acid, sodium benzenesulphonate, sodium chloride, and a small quantity of sodium sulphate are also produced. Benzylphenylsulphone is formed, together with numerous unimportant products, when benzotrichloride and sodium benzenesulphinate are heated together in alcoholic solution. F. S. K.

Iodophenolsulphonic Acids. By F. Kehrmann (*J. pr. Chem.* [2], **37**, 334—342; compare this vol., p. 595).—When the potassium salt of the diiodophenolsulphonic acid previously described is shaken with nitric acid of sp. gr. 1·52, iodine separates, and the solution contains what is probably a dinitrophenolsulphonic acid, the potassium salt of which forms reddish-yellow crystals readily soluble in water. If, however, the iodine is filtered off and the solution boiled for a few minutes, it gives on cooling pure picric acid; hence the diiodophenolsulphonic acid must have the constitution

$$OH{\cdot}C_6H_2I_2{\cdot}SO_3H\ [OH : I_2 : SO_3H = 1 : 2 : 6 : 4].$$

Di-iodoquinone, $C_6H_2O_2I_2$ [$O_2 : I_2 = 1 : 4 : 2 : 6$], is obtained when di-iodoparaphenolsulphonic acid is oxidised with chromic acid. It forms reddish-yellow scales, melts at 179—180° (uncorr.), and sublimes readily. The corresponding quinol crystallises in colourless needles melting at 144—145° (compare Seifert, Abstr., 1884, 431).

Orthoiodocresolsulphonic acid,

$$OH{\cdot}C_6H_2MeI{\cdot}SO_3H\ [OH : Me : SO_3H : I = 1 : 2 : 4 : 6],$$

crystallises in large plates containing 3 mols. H_2O. The potassium and barium salts are described. With nitric acid of sp. gr. 1·52, it

yields a dinitrocresol [OH : Me : $(NO_2)_2$ = 1 : 2 : 4 : 6] melting at 85—86°, and on oxidation with chromic acid an iodomethylquinone [Me : O_2 : I = 2 : 1 : 4 : 6], crystallising in golden-red needles and melting at 115°, is obtained.

Under some circumstances when potassium orthocresolparasulphonic acid is iodated, besides the acid above described, an acid of the constitution $OH{\cdot}C_6H_2MeI{\cdot}SO_3H$ [Me : OH : SO_3H : I = 2 : 1 : 6 : 4] is obtained. With nitric acid of sp. gr. 1·52, it forms the dinitrocresol melting at 85—86°, but on oxidation with chromic acid suffers decomposition with liberation of iodine.

On sulphonating thymol and treating the sulphonic acid so formed with iodine, a monoiodothymolsulphonic acid is obtained, which on oxidation with chromic acid furnishes a moniodopropylmethyl quinone, and therefore contains the sulphonic acid group in the para-position to the hydroxyl. G. T. M.

Di-iodophenolsulphonic Acid. By F. Kehrmann (*J. pr. Chem.* [2], **37**, 359—360).—This is a controversial paper in answer to a recent one by Ostermayer (this vol., p. 596).

Splitting of the Naphthalene and Benzene Rings by Oxidation. By R. Henriques (*Ber.*, **21**, 1607—1620).—When an aqueous solution of sodium α-naphthoxide is treated with potassium permanganate so long as the latter is decolorised, orthocarboxylphenylglyoxylic acid, $COOH{\cdot}C_6H_4{\cdot}CO{\cdot}COOH$ (Zincke and Breuer, Abstr., 1885, 270, and Scherks, *ibid.*, 533), is obtained, but scarcely any phthalic acid is formed, as when the oxidation takes place in acid solution. When heated with alkaline permanganate solution, phthalic acid is slowly formed. *Anhydrophenylhydrazinorthocarboxylphenylglyoxylic acid*, $CO<\begin{matrix}C_6H_4\\N_2Ph\end{matrix}>C{\cdot}COOH$, is precipitated in yellow flakes when a solution of the acid is warmed with phenylhydrazine hydrochloride, and crystallises in colourless needles melting at 214—215°. It is sparingly soluble in water and ether, rather readily soluble in hot alcohol and hot glacial acetic acid. Alkalis and alkaline carbonates dissolve it readily. The *methyl* salt, prepared by heating the hydrazine compound with methyl alcohol and methyl iodide, forms readily soluble needles which melt at 114°. When the free acid is heated above its melting point, it is converted into the internal anhydride, $C_{14}H_{10}N_2O$ (Racine, Abstr., 1887, 951), which melts at 106—107°. This when boiled for a long time yields phthalanil.

When α-naphthol (100 grams) is oxidised with potassium permanganate (500 grams) in presence of soda (28 grams), filtered from manganese dioxide, and acidified with sulphuric acid, 95 grams of crude phthalic acid, together with an acid of the formula $C_{20}H_{14}O_8$ are obtained. The latter separates in yellow flakes on adding acid, and is purified by boiling it several times with glacial acetic acid and with alcohol. It forms a very insoluble, white powder, which melts at 246°. The *sodium* and *barium* salts are very readily soluble; the latter crystallises well. When treated with potassium permanganate in alkaline solution, it yields carboxyphenylglyoxylic acid. When

boiled with an excess of phenylhydrazine, the compound $C_{26}H_{18}N_2O_6$ is obtained. This crystallises in small needles melting at 175° with decomposition, very readily soluble in alcohol, less in benzene. When the acid $C_{20}H_{14}O_8$ is heated at its melting point, it gives off carbonic anhydride and is converted into diphthalyl and a new acid of the formula $C_{19}H_{10}O_6$; this melts at 162—163°.

If in the oxidation of α-naphthol the temperature is not kept below 35—40°, and the product after being acidified is treated with phenylhydrazine, a mixed powder is obtained containing anilphthalimide and Hötte's phenylhydrazide (Abstr., 1887, 669).

When α- and β-naphthalenesulphonic acids are oxidised with potassium permanganate, they also yield orthocarboxylphenylglyoxylic acid.

When a dilute aqueous solution of phenol is gradually treated with potassium permanganate, a considerable amount of oxalic acid is formed; when a larger amount of permanganate (330 grams to 50 grams of phenol) is used, a small quantity of salicylic acid is produced.

N. H. M.

Action of Quinonedichlorimide on β-Naphthylamine. By R. Nietzki and R. Otto (*Ber.*, **21**, 1598—1602).—When quinone dichlorimide is added to β-naphthylamine dissolved in 5 to 6 parts of alcohol, the solution acquires an intense red colour, and on adding water the colour changes to yellow, and a separation of yellowish-brown flakes takes place. The compound so formed is a eurhodine of the formula $C_{10}H_6\langle\begin{smallmatrix}N\\|\\N\end{smallmatrix}\rangle C_6H_3\cdot NH_2$. It crystallises from benzene in dark yellow needles, rather sparingly soluble in alcohol and benzene, readily soluble in aniline. The alcoholic solution shows a strong fluorescence. Hydrochloric and sulphuric acids dissolve it with formation of intense magenta and reddish-brown coloured solutions respectively. When the solution in 60 parts of alcohol is treated with sulphuric acid and sodium nitrite, Witt's naphthophenazine is formed.

Quinonedichlorimide reacts with phenyl-β-naphthylamine in alcoholic solution with formation of the *compound* $C_{10}H_6\langle\begin{smallmatrix}N\\|\\NPhCl\end{smallmatrix}\rangle C_6H_3\cdot NH_2$. The red solution is precipitated with zinc chloride. It is purified by means of the nitrate. It forms a blue-violet precipitate soluble in alcohol and ether; the solutions are also violet and are not fluorescent. The *nitrate* crystallises in slender needles. The dye has a striking resemblance to that obtained by removing an amido-group from safranin. It is dissolved by strong sulphuric acid with blackish-violet colour, which changes on addition of water to a dirty green, then to orange, and finally to carmine-red.

The assumption of the presence of a phenylazonium-group is supported by the fact that by the introduction of phenyl into the but feebly basic eurhodine a rather strong base is formed which yields salts not decomposed by alkaline carbonates.

N. H. M.

Nitro-derivatives of Acenaphthene. By F. Quincke (*Ber.*, **21**, 1454—1463; compare Abstr., 1887, 592). —When acenaphthene

(80 grams) dissolved in acetic acid (1 kilo.) is treated with nitric acid (50 c.c.) free from lower oxides of nitrogen, it yields a mixture of nitracenaphthene (50 grams) and dinitracenaphthene (20 grams), from which the former can be extracted by digestion with light petroleum. *Dinitracenaphthene*, $C_{12}H_8(NO_2)_2$, crystallises in yellow needles, melts at 206° with carbonisation, and is probably identical with Berthelot's derivative obtained by the action of fuming nitric acid (*Bull. Soc. Chim.*, **8**, 250).

When reduced with tin and hydrochloric acid, nitroacenaphthene is converted into amidacenaphthene. The product, however, is not pure, since a portion is further reduced to acenaphthene, which unites with the amine to form a compound crystallising from alcohol with a constant melting point of 96°, whilst amidacenaphthene itself crystallises in needles melting at 108°. The *hydrochloride*, $C_{12}H_9{\cdot}NH_2,HCl$, crystallises in slender needles; the *platinochloride*,

$$(C_{12}H_9{\cdot}NH_2)_2,H_2PtCl_6,$$

crystallises in deliquescent, reddish-yellow needles; the *stannochloride*, $C_{12}H_9{\cdot}NH_2,HSnCl_3$, crystallises in brownish-red, prismatic forms, and the *picrate*, $C_{12}H_9{\cdot}NH_2,C_6H_2(NO_2)_3{\cdot}OH$, forms yellow crystals. The *acetyl*-derivative, $C_{12}H_9{\cdot}NHAc$, crystallises from alcohol in dark yellow scales, and melts at 176°; the *diacetyl*-derivative, $C_{12}H_9{\cdot}NAc_2$, crystallises from alcohol in bright brown spangles and melts at 122°; the *benzoyl*-derivative, $C_{12}H_9{\cdot}NHBz$, crystallises from alcohol in silky yellowish needles and melts at 210°; the *thiocarbamide*, $CS(NH{\cdot}C_{12}H_9)_2$, crystallises from toluene in slender, pale yellow or violet, asbestos-like needles and melts at 192°, and the *thiocarbimide*, $C_{12}H_9NCS$, crystallises from alcohol in brown lustrous scales and melts at 96°.

Diamidacenaphthene is obtained from dinitracenaphthene by reduction either with tin and hydrochloric acid, or with hydriodic acid and phosphorus at 100°, but could not be separated in a pure form owing to the ease with which it decomposes. The salts are likewise very unstable; the *picrate* crystallises in yellow needles; the *hydrochloride*, $C_{12}H_8(NH_2)_2,2HCl$, and *hydriodide*, $C_{12}H_8(NH_2)_2,2HI$, were also prepared.

When oxidised with potassium chromate and dilute sulphuric acid, with potassium permanganate in alkaline solution, or best with nitric acid (sp. gr. = 1·2), nitracenaphthene is converted into nitronaphthalic acid and nitro-γ-naphthaquinone. A better yield, however, is obtained by heating acenaphthene (30 grams) with nitric acid of sp. gr. = 1·2 (600 c.c.) in a reflux apparatus for three hours, whereby both the acid (2 grams) and the quinone (15 grams) are formed. *Nitronaphthalic acid*, $NO_2{\cdot}C_{10}H_5(COOH)_2$, crystallises in slender yellow needles, is sparingly soluble in alcohol, ether, and light petroleum, and decomposes at 140—150° with the formation of the *anhydride*, $NO_2{\cdot}C_{10}H_5{:}C_2O_2{:}O$, which melts at 220°. The salts are readily soluble in water; the *calcium* salt, with 1 mol. H_2O, crystallises in yellow scales, and the *ammonium* salt, with 1 mol. H_2O, in yellow needles. *Nitro-γ-naphthaquinone*, $C_{10}H_5O_2{\cdot}NO_2$, crystallises in yellowish-red needles, melts at 208°, and is soluble in alcohol and acetic acid. The *anilide*, $NO_2{\cdot}C_{10}H_4O_2{\cdot}NHPh$, crystallises in dark

violet needles, melts at 128° with decomposition, and is sparingly soluble in all the ordinary solvents except acetic acid; the *diphenylamine*-derivative, $NO_2 \cdot C_{10}H_4O_2 \cdot NPh_2$, forms green flocks, melts at 80° with decomposition, and is readily soluble in alcohol, acetic acid, and light petroleum, but sparingly soluble in ether.

W. P. W.

Molecular Refraction as a Means of Determining the Constitution of Members of the Terpene-group. By O. Wallach (*Annalen*, **245**, 191—213).—As in some cases the terpenes which Brühl used in his research on molecular refraction (this vol., pp. 377 and 494) were impure, the author has re-determined the index of refraction with terpenes of undoubted purity with the following results:—

	n_c.	$\frac{(n^2-1)p}{(n^2+2)d}$	Calculated refraction equivalent.
Cineol, $C_{10}H_{18}O$	1·4559	45·16	45·10
Terpineol, $C_{10}H_{18}O$	1·48084	46·82	46·88
Dipentene, $C_{10}H_{16}$	1·47308	45·10	45·00
Sylvestrene, $C_{10}H_{16}$	1·47468	45·08	45·00

W. C. W.

Action of Acids and Anhydrides on Terpilenols. By J. Lafont (*Compt. rend.*, **106**, 1170—1171).—Terpilenol mixed with an equivalent quantity of formic acid slowly undergoes etherification at the ordinary temperature, but the change is always far from complete. At 100°, or with equal weights of terpilenol and acid, the terpilenol splits up into water and terpilene, the latter becoming partially polymerised. With acetic acid at the ordinary temperature, terpilenol undergoes etherification very slowly, and the reaction is far from complete. At 100—150° the change is more rapid, and at the higher temperature it may amount to 5 per cent.

Terpilenol can, however, be readily and completely converted into the acetate, $C_{10}H_{18}.C_2H_4O_2$, by heating it with a slight excess of acetic anhydride in sealed tubes at 100° for 24 hours. When treated with alcoholic potash at 100°, the acetate yields the original terpilenol.

Precisely the same results were obtained with terpilenols from two different sources.

When terpilenol from terpin is treated with aqueous chromic acid, no aldehyde and no ketone is formed, but acetic acid is obtained in small quantity and terpene is produced by dehydration. The chromic acid partially destroys the molecule. From these results it follows that terpilenols have many of the peculiarities of tertiary alcohols.

C. H. B.

Camphor from the Ethereal Oil of Ledum Palustre. By B. Rizza (*J. Russ. Chem. Soc.*, 1887, 319—325).—A. Gorboff has examined the posthumous papers of B. Rizza, and in his diary finds data according to which the camphor from *Ledum palustre* has the formula $C_{15}H_{26}O$. On heating the camphor with acetic anhydride, two layers are obtained, of which the lower consists of acetic acid, whilst the upper contains a hydrocarbon of the composition $C_{15}H_{24}$, belonging to the class of sesquiterpenes $(C_5H_8)_3$; this is a liquid of

sp. gr. 0·9349 at 0°, and 0·9237 at 19°, and boils at 264°. It combines with bromine. The camphor is regarded by the author as a sesquiterpene hydrate. The oil of *Ledum palustre* seems to be identical with the above hydrocarbon, as after drying it distils in a current of hydrogen at 260—270°. B. B.

Hydrocarbons in Resin Oils. By A. Renard (*Compt. rend.*, **106**, 1086—1087).—That portion of resin oil which is not attacked by strong sulphuric acid yields a mixture of two hydrocarbons boiling at 330—350°, which cannot be separated by fractionation. The portion boiling at 340—350° contains diterebenthylene identical with that obtained by the action of heat on diterebenthyl dibromide. The portion boiling at 330—340° also contains diterebenthylene, but consists mainly of a new hydrocarbon; this is isolated by two or three successive treatments with sulphuric acid or with fuming nitric acid, which converts the diterebenthylene into either a sulphonic derivative soluble in water or a nitro-derivative soluble in nitric acid. The unattacked portion is washed with sodium hydroxide and rectified over sodium.

The hydrocarbon thus obtained has the composition $C_{20}H_{36}$, and is a colourless non-fluorescent oily liquid, which boils at 330—335°; sp. gr. at 12° = 0·9362; rotatory power of a column 10 cm. in length for D = −2°. It does not alter when exposed to air, and is not affected by fuming sulphuric, nitric, or hydrochloric acid, nor by bromine at the ordinary temperature. It may be regarded as didecene, a polymeride of decene, $C_{10}H_{18}$.

From these and previous results, it appears that resin oils contain 80 per cent. of diterebenthyl, 10 per cent. of diterebenthylene, and 10 per cent. of didecene. C. H. B.

Shellac. By R. Benedikt and E. Ehrlich (*Monatsh.*, **9**, 157—164).—When shellac, previously freed from fat by boiling with sodium carbonate, is boiled with caustic alkalis for two hours, and the cold solution acidified with sulphuric acid, about 70 per cent. of a viscous, liquid shellac is precipitated. The product is extracted with ether, and purified by means of the magnesium salt ($C_{46}H_{70}Mg_2O_{13}$). It is a thick, viscous liquid, which becomes mobile when heated, and is only very sparingly soluble in boiling water, but dissolves readily in alcohol and ether. The alcoholic solution is precipitated by water. When heated, water is evolved and on cooling a solid mass very similar to ordinary shellac is obtained. The acid value of liquid shellac is nearly three times as great as that of ordinary shellac, 1 gram requiring 0·204 gram of potash for complete saturation. From this datum and from the elementary analysis, the formula of liquid shellac is probably $C_{46}H_{72}O_{12}$. A mixture of ordinary and liquid shellac is obtained by boiling two portions of shellac, one with sodium carbonate, the other with soda, separating the wax and acidifying the cold mixed solutions with acetic acid. It is a plastic resin which when free from acid retains its plastic condition for a considerable time, but after several months gradually begins to harden at the surface. The alkaline-earth salts of liquid shellac are

soluble in cold water in all proportions, they are precipitated as thick liquids when the solution is boiled, but redissolve completely on cooling. When an aqueous solution is evaporated over sulphuric acid, a completely transparent residue is obtained, which after some time becomes opaque. These salts are very brittle, and even in the dry state are very readily soluble in cold water. The *barium* salt is obtained by neutralising an alcoholic solution with baryta-water. A solution of the magnesium salt gives with lead, silver, and zinc salts white precipitates which form resinous masses when warmed.

Shellac freed from wax yields 20 per cent. of azelaïc acid when boiled with potash and potassium permanganate, products smelling like butyric acid being also formed. Shellac is completely converted into azelaïc acid and fatty acids by potassium permanganate, when the residual resin is again boiled with the permanganate, and the process repeated until the whole is oxidised. F. S. K.

Apiole. By G. Ciamician and P. Silber (*Ber.*, **21**, 1621—1633). —*Iso-apiole*, $C_{12}H_{14}O_4$, is prepared by heating 25 grams of apiole with 50 grams of potash and 250 c.c. of absolute alcohol for 10 to 15 hours on a water-bath. The product is cooled, poured into 1 litre of water and filtered. The crystals are pressed between filter-paper and crystallised from alcohol. It forms large plates, melts at 55—56°, and boils at 303—304°, and is readily soluble except in water. The yield is 70 to 75 per cent.

Apiolic acid, $C_{10}H_{10}O_6$, is obtained by gradually adding a solution of 32 grams of potassium permanganate in 1600 c.c. of water to 8 grams of iso-apiole in 800 c.c. of boiling water containing potash. The mixture is afterwards heated in a water-bath for one hour. The yield is 37 to 38 per cent. A small quantity of apiolaldehyde melting at 102° (Ginsberg, this vol., p. 722) is also formed. Apiolic acid crystallises from water in needles melting at 175°, soluble in ether, hot alcohol, hot glacial acetic acid, benzene, and ethyl acetate. The *silver salt*, $C_{10}H_9AgO_6$, crystallises in large needles, the *calcium salt* in prisms of a glassy lustre. The *methyl salt* melts at 71—72°, and dissolves in ether, alcohol, and glacial acetic acid, sparingly in hot water. The same acid is formed in small quantity in the oxidation of apiole with potassium permanganate, the chief product being a neutral compound of the formula $C_{12}H_{16}O_6$ (this vol., p. 606).

Apiolaldehyde, $C_{10}H_{10}O_5$, (Ginsberg, *loc. cit.*) is prepared by gradually treating 2 grams of iso-apiole with a mixture of 10 grams of potassium dichromate with 200 grams of dilute sulphuric acid (1 : 10) and boiling for three hours. It is then steam-distilled to remove acetaldehyde and acetic acid, which are also formed, filtered, and the crystals which separate on cooling crystallised from alcohol. It is readily purified by means of the *hydrogen sodium sulphite compound*. The aldehyde can be obtained from apiole but the yield is not at all good. The *oxime*, $C_{10}H_{10}O_4{:}N{\cdot}OH$, crystallises in long needles, melts at 160—161°, dissolves sparingly in hot water, readily in ether, hot alcohol, and acetic acid; it dissolves in sulphuric acid with yellow colour which changes to olive-green when the solution is slightly warmed.

When apiolaldehyde is oxidised with permanganate, apiolic acid melting at 175° is formed almost quantitatively. When apiolaldehyde (1 gram) is dissolved in glacial acetic acid (10 c.c.), and treated with cold nitric acid (sp. gr. 1·35, 40 grams), a *nitro-derivative*, $C_7H_7NO_5$, is obtained. This crystallises from alcohol in yellow needles which melt at 137—138°.

When apiolic acid is heated with dilute sulphuric acid at 140° for five hours, it is converted into *apione*, $C_9H_{10}O_4$, and carbonic anhydride. The product is steam-distilled, and the slender, white needles so obtained crystallised from alcohol. It melts at 79°, dissolves readily in ether, acetic acid, and boiling alcohol, and is insoluble in water.

A bromine-derivative, probably *dibromapione*, is formed when apiolic acid or the aldehyde is treated with bromine. It melts at 99—100°.

The physiological properties of iso-apiole and of apiole are described. N. H. M.

Picrotoxin. By B. v. d. Marck (*Arch. Pharm.* [3], **26**, 269, from *Nieuw. Tijdsch. Pharm. Nederland.*, 1888, 25).—In opposition to the views of others, the author recommends light petroleum for the extraction of picrotoxin from the pulverised fruit of *Anamista coccula*. On distilling off the solvent, a crystalline product is obtained, soluble in acidified water, with the exception of some chlorophyll and resin. The filtrate on evaporation, gives the picrotoxin in pale-yellowish crystals, which by repeated recrystallisation become snow-white. J. T.

Ophioxylin. By W. Bettink (*Arch. Pharm.* [3], **26**, 268—269, from *Nieuw. Tijdschr. Pharm. Nederland.*, 1888, 1).—This crystallisable compound, obtained from the root of *Ophioxylon serpentinum*, forms orange-yellow crystals of the tetragonal system; it has a sharp, burning taste, is sparingly soluble in water, more soluble in alcohol, and very soluble in chloroform, benzene, and carbon bisulphide. Its elementary analysis gives the formula $C_{16}H_{13}O_6$ or $C_{48}H_{39}O_{18}$. In most of its properties and reactions, it agrees with juglone, but it melts at 71·8°, whilst juglone melts at 151—154°. J. T.

Crystalline Arrow-poison of the Comalis, extracted from the Wood of the Ouabaïo.—By Arnaud (*Compt. rend.*, **106**, 1011—1014).—The Comalis on the East Coast of Africa prepare an arrow-poison from the aqueous extract of the wood, and especially of the root of the Ouabaïo, a tree which is closely related to although not identical with *Carissa schimperii*, a native of Abyssinia.

A concentrated extract of the wood in warm water was prepared and precipitated with lead acetate, the filtrate treated with hydrogen sulphide, and the second filtrate boiled and afterwards concentrated in a vacuum. The concentrated solution was mixed with six times its volume of alcohol of 85°, boiled, allowed to cool in shallow vessels, and the crystals thus obtained purified by recrystallisation first from alcohol of 85° and afterwards from water.

The *ouabaïn* thus obtained contains no nitrogen and has the composition $C_{30}H_{46}O_{12}$. It forms thin, white, nacreous lamellæ with no

taste, no smell, and a neutral reaction. It is almost insoluble in cold water, but is readily soluble in boiling water, with a great tendency to form supersaturated solutions, and also dissolves readily in moderately concentrated alcohol, but is almost insoluble in absolute alcohol and insoluble in ether or chloroform. At 180°, it becomes pasty with slight decomposition, and at 200° it is completely melted. A warm aqueous solution has a rotatory power $[\alpha]_D = -34°$. A concentrated aqueous solution gives a precipitate with tannin. When boiled with dilute acids, it yields a reducing sugar, and hence it would seem that ouabaïn is a glucoside.

When ouabaïn crystallises from an aqueous solution, it contains 7 mols. H_2O which is not completely expelled below 130°.

When boiled with barium hydroxide, ouabaïn yields a barium-derivative which when dried at 100° has the composition $Ba(C_{30}H_{47}O_{13})_2$.

Ouabaïn has no toxic effect when introduced into the stomach, but when introduced by subcutaneous or intravenous injection, it acts on the heart and produces death. 2 mgrams. will kill a dog of 12 kilos. in a few minutes. C. H. B.

Bases Formed by the Action of Hydrochloric Acid on Pyrrolines. By M. Dennstedt and J. Zimmermann (*Ber.*, **21**, 1478—1481).—The hydrochloride of a base which is obtained when hydrochloric acid is passed into an ethereal solution of pyrroline (Abstr., 1887, 598) has the formula $(C_4NH_5)_3,HCl$. It is soluble in water and alcohol, but cannot be crystallised from either solvent. The *platinochloride* and *picrate* are both unstable. Potassium chromate, ferrocyanide and ferricyanide give brown, amorphous precipitates. The sparingly soluble *hydriodide* is precipitated in white crystals by adding potassium iodide, and the free base $(C_4NH_5)_3$ is obtained in the form of a white, amorphous precipitate on adding ammonia to an aqueous solution of the hydrochloride. The base is decomposed not only when heated at a relatively low temperature, but also when heated with methyl iodide, or when boiled with acetic anhydride and sodium acetate.

The crystalline salt obtained when hydrochloric acid is passed into an ethereal solution of isopropylpyrroline (*loc. cit.*) has the formula $(C_7NH_{11})_2HCl$. The *picrate*, $(C_7NH_{11})_2,C_6H_3N_3O_7$, crystallises from alcohol in long, brown, shining needles melting at 146°. The free base is a heavy, slightly yellow oil which boils at 285—290° and smells something like turpentine. When heated quickly, it can be distilled with partial decomposition, and on exposure to the air it is gradually converted into the isomeric isopropylpyrroline.

F. S. K.

Reduction of Pyrrolinephthalide. By M. Dennstedt and J. Zimmermann (*Ber.*, **21**, 1554—1556).—*Pyrrolinehydrophthalide*,

$$C_4NH_3{:}C{<}\begin{matrix}-O-\\ C_6H_4\end{matrix}{>}CH{\cdot}OH,$$

is prepared by gradually adding 8 per cent. sodium amalgam to 2 to 3 grams of finely powdered pyrrolinephthalide suspended in 250 c.c. of water; after two to three days, it is filtered through asbestos, very

slightly acidified with quite dilute sulphuric acid and several times quickly extracted with ether. It crystallises from alcohol in needles melting at 118°, and is readily soluble in the usual solvents. The crystals as well as the solutions of the substance soon become reddish or green. When the ethereal solution is evaporated very gradually, well-formed, lustrous, rhombohedro-tetartohedric prisms are obtained; $a:c = 1:0{\cdot}8491$. The *silver salt* is an unstable, reddish, crystalline substance, which when dried, even in the dark, decomposes with explosive violence. N. H. M.

Action of Pyridine on Metallic Salts. By W. Lang (*Ber.*, **21**, 1578—1588).—*Zinc chloride pyridine*, $ZnCl_2,2C_5NH_5$, is prepared by treating an aqueous solution of zinc chloride with an excess of pyridine, when it separates as a voluminous, white precipitate; its formation is accompanied by development of heat. It crystallises from water in long, white needles of a silky lustre which consist of fibrous aggregates. When washed or boiled repeatedly with fresh quantities of water, it decomposes with liberation of pyridine and formation of a basic zinc chloride. If the same water is used, the decomposition is limited. The compound is rather stable when exposed to air, and is only slowly decomposed at 130° to 160°. It is possible to free alcohol from pyridine which it may contain by adding zinc chloride. The *hydrochloride*, $ZnCl_2 2C_5NH_5(HCl)_2$, forms groups of white, lustrous needles.

Cupric chloride pyridine, $CuCl_2,2C_5NH_5$, is precipitated in splendid greenish-needles of a silky lustre, when pyridine is added to an alcoholic solution of cupric chloride. It is soluble in pyridine, in water containing pyridine, and in aqueous ammonia; it is decomposed by water, but only so long as the amount of pyridine liberated does not reach a certain point. At the ordinary temperature it is stable and loses only traces of pyridine when heated at 100°; it melts at 180—190°. The *hydrochloride* crystallises from water in dark-yellow prisms.

Cupric sulphate pyridine, $CuSO_4,C_5NH_5 + 3H_2O$, crystallises in sky-blue needles of silky lustre; when heated at 100°, it gives up water without losing its lustre; the anhydrous salt, probably identical with that obtained by Jörgensen (Abstr., 1886, 857), recovers its blue colour when exposed to air.

Cuprous chloride pyridine, $Cu_2Cl_2,4C_5NH_5$, obtained by adding solid cuprous chloride to pyridine, crystallises in greenish-gold-coloured, transparent octahedra, which quickly decompose when exposed to air. When a large excess of pyridine is used, the *compound* $Cu_2Cl_2,6C_5NH_5$ separates in long, greenish-yellow, transparent needles which lose pyridine more quickly than the regular crystals. The solution absorbs carbon monoxide just as readily as ammoniacal cuprous chloride solution.

Cadmium chloride pyridine, $CdCl_2,2C_5NH_5$, crystallises from water (which decomposes it partly) in needles.

Cadmium iodide pyridine, $CdI_2,2C_5NH_5$, forms long, white needles of a silky lustre.

A table is given showing the amount of pyridine lost by the above compounds at temperatures varying from 100° to 160°.

Mercuric chloride pyridine, $HgCl_2,C_5NH_5$, is rather readily decomposed by water; it is stable when exposed to air, but is decomposed at 100°.

With mercurous salts, no derivatives could be obtained; mercury separates and the mercuric compounds are formed.

Calcium chloride pyridine, $CaCl_2,3C_5NH_5$, is a loose, white powder. Compounds of pyridine with cobaltous chloride, ferrous sulphate, and nickel sulphate are also described. N. H. M.

Condensation of Ethyl Tetramethylphenylamidocrotonate. By M. Conrad and L. Limpach (*Ber.*, **21**, 1655—1657).—*Ethyl tetramethylphenylamidocrotonate*, $C_6HMe_4\cdot NH\cdot CMe{:}CH\cdot COOEt$, is obtained by the action of tetramethylamidobenzene [$NH_2 : Me_4 = 1:2:3:4:6$], prepared from pseudocumidine (Nölting and Baumann, Abstr., 1885, 893; compare also Limpach, this vol., p. 464) on ethyl acetoacetate. After two days, the solid portion of the product is crystallised from ether and alcohol. It forms large, white prisms, melting at 101°.

Tetramethylphenyllutidoncarboxylic acid,

$$C_6HMe_4\cdot N<\begin{matrix}CMe{:}C(COOH)\\ -CMe{:}CH\cdot CO-\end{matrix}>,$$

is formed (as ethyl salt) together with tetramethylamidobenzene and ethyl alcohol when ethyl tetramethylphenylamidocrotonate is heated for some minutes at 280—285°. The product is heated in a reflux apparatus with alcoholic potash. The acid is readily soluble in alcohol and ether, melts at 145° and decomposes carbonates. N. H. M.

Azo-dyes of the Hydroxyquinolines. By J. Mathëus (*Ber.*, **21**, 1642—1647).—*Benzeneazoparahydroxyquinolinesulphonic acid*,

$$OH\cdot C_9NH_5\cdot N_2\cdot C_6H_4\cdot SO_3H,$$

prepared from diazobenzene sulphochloride and parahydroxyquinoline, crystallises in small, orange-red needles, soluble in hot water and cold dilute alkali. The *sodium salt* forms red needles.

Benzeneazoparahydroxyquinoline, $OH\cdot C_9NH_5\cdot N_2\cdot Ph$, obtained from diazobenzene chloride and parahydroxyquinoline, crystallises from absolute alcohol in long, orange-red needles, soluble in ether and benzene, insoluble in water; the *sodium salt* forms red needles.

Paratolueneazoparahydroxyquinoline, $OH\cdot C_9NH_5\cdot N_2\cdot C_6H_4Me$, from diazoparatoluene chloride, crystallises from alcohol in wide, red needles, soluble in ether and benzene. The *sodium salt* forms dark-red needles.

Parabromobenzeneazoparahydroxyquinoline, $OH\cdot C_9NH_5\cdot N_2\cdot C_6H_4Br$, crystallises from chloroform in bright-red, matted needles. It dissolves sparingly in alcohol, readily in chloroform and in alcoholic soda.

β-Naphthaleneazoparahydroxyquinoline, $OH\cdot C_9NH_5\cdot N_2\cdot C_{10}H_7$, crystallises from absolute alcohol in small, dark-red needles. The *sodium salt* has a still darker red colour.

Benzeneazo-orthohydroxyquinoline, $C_{15}H_{11}N_3O$, crystallises from alcohol in long, brownish-yellow, lustrous needles, soluble in ether, in chloroform, and in alcoholic potash. The *sodium salt* is yellowish-brown.

Paratolueneazo-orthohydroxyquinoline, $C_{16}H_{13}N_3O$, crystallises from chloroform in yellowish-brown plates of a metallic lustre.

Parabromobenzeneazo-orthohydroxyquinoline, $C_{15}H_{10}BrN_3O$, is precipitated by hydrochloric acid from its solution in alcoholic soda in slender, gold-coloured needles soluble in absolute alcohol and ether.

β-Naphthaleneazo-orthohydroxyquinoline, $OH{\cdot}C_9NH_5{\cdot}N_2{\cdot}C_{10}H_7$, forms reddish-brown needles, soluble in ether, absolute alcohol, chloroform, and alcoholic soda.

Amidoparahydroxyquinoline, $OH{\cdot}C_9NH_5{\cdot}NH_2$, is formed together with sulphanilic acid when a mixture of benzeneazoparahydroxyquinoline-sulphonic acid and an excess of stannous chloride is treated with hydrochloric acid. It is purified by carefully neutralising the rather concentrated solution of the hydrochloride with solid sodium carbonate, when the free base separates in slender needles which are dried on tiles. It dissolves readily in absolute alcohol, sparingly in ether, benzene, and chloroform. Alkali solutions dissolve it readily with a green colour which quickly changes to brown. It crystallises from water with 2 mols. H_2O. The hydrochloride forms red prisms readily soluble in water; the sulphate crystallises in reddish-yellow prisms soluble in hot water; the *picrate* separates from its alcoholic solution in dark-red plates; the stannochloride forms lustrous, yellow plates, readily soluble in cold water. (Compare Abstr., 1884, 1370.)

N. H. M.

Lepidine-derivatives. By B. Heymann and W. Koenigs (*Ber.*, **21**, 1424—1430).—When lepidine [Me = 4'] is treated with benzaldehyde and zinc chloride, it is converted into benzylidenelepidine, which is reduced to *benzyllepidine*, $C_9NH_6{\cdot}CH_2{\cdot}CH_2Ph$, by boiling for 10 hours with a mixture of acetic acid, amorphous phosphorus, and hydriodic acid (saturated at 0°). This crystallises well from alcohol and light petroleum, and melts at 100—101°.

Parahydroxybenzylidenelepidine,

$$C_9NH_6{\cdot}CH{:}CH{\cdot}C_6H_4{\cdot}OH \quad [CH:OH = 1:4],$$

is obtained by heating lepidine (2 grams) with parahydroxybenzaldehyde (1·7 grams), and finely powdered potassium hydrogen sulphate (5 grams) at 150—160° for eight hours. It crystallises from absolute alcohol in anhydrous, yellow needles or prisms, melts at 252—253°, and is sparingly soluble in dilute aqueous soda. Its *hydrochloride* crystallises in fiery-red needles, and is sparingly soluble in alcohol and water. On reduction, the base yields *parahydroxybenzyllepidine*, $C_9NH_6{\cdot}CH_2{\cdot}CH_2{\cdot}C_6H_4{\cdot}OH$, which melts at 179—180°, is crystalline, and closely resembles homo-apocinchine (this vol., p. 72) in its solubility, melting point, and crystalline appearance: the hydrobromides of the two bases also crystallise in similar forms, and show a like solubility in water, but the hydrobromide of the former melts at about 250°, whereas that of the latter melts at 220—222°. The crystals of the two bases could not be obtained in a suitable form for crystallographic measurement.

Orthohydroxybenzylidenelepidine,

$$C_9NH_6{\cdot}CH{:}CH{\cdot}C_6H_4{\cdot}OH \quad [CH:OH = 1:2],$$

prepared in like manner from orthohydroxybenzaldehyde, crystallises in slender, yellow forms, melts at 219°, and dissolves more readily in alcohol than the para-compound. The *hydrochloride* is a reddish-yellow salt, and is sparingly soluble in water and alcohol.

Metanitrobenzylidenelepidine, $C_9NH_6{\cdot}CH{:}CH{\cdot}C_6H_4{\cdot}NO_2$ [$CH : NO_2 = 1 : 3$], is obtained by heating lepidine (2 grams) with metanitrobenzaldehyde (2·1 grams) and potassium hydrogen sulphate (5 grams) at 150—160° for eight hours. It crystallises from alcohol in pale greenish-yellow, concentrically grouped needles, melts at 135—136°, and is sparingly soluble in cold alcohol. The *hydrochloride* forms yellow flocks, and is very sparingly soluble in water and alcohol.

Benzylquinaldine, $C_9NH_6{\cdot}CH_2{\cdot}CH_2Ph$, obtained from quinaldine by a method similar to that employed for the preparation of benzyllepidine, forms colourless crystals, melts at 30°, and is readily soluble in benzene and light petroleum. The *picrate* crystallises in bright yellow prisms, melts at 130°, and is sparingly soluble in hot alcohol.

W. P. W.

Synthesis of Dihydroxyquinaldine-derivatives. By M. Conrad and L. Limpach (*Ber.*, **21**, 1649—1654).—*Ethyl paramethoxyphenylamidocrotonate*, $C_{13}H_{17}NO_3$, is prepared by mixing an ethereal solution of paranisidine (m. p. 56°) with ethyl acetoacetate. After 24 to 28 hours, the ether is evaporated off, and the ethyl salt remains as a crystalline mass. It crystallises from alcohol in large prisms melting at 46°.

Paramethoxy-γ-hydroxyquinaldine,

$$OH{\cdot}C_9NH_3Me{\cdot}OMe\ [Me : OH : OMe = 2' : 4' : 3],$$

is obtained by heating the above ethyl salt for a few minutes at 260°; the yield is 37 per cent. of the theoretical. When heated quickly, it melts at 290° with only slight decomposition. It is sparingly soluble in boiling water. The *hydrochloride* crystallises in long needles, readily soluble in hot water; the *platinochloride* forms long, lustrous, orange-yellow prisms readily soluble in hot water; the *sulphate* crystallises in needles soluble in about 170 parts of cold water.

Paramethoxy-γ-chloroquinaldine, $C_{11}H_{10}NOCl$, is prepared by heating the dry hydroxy-derivative with twice the amount of phosphorus oxychloride in a reflux apparatus first at 100° and afterwards at 140°, as long as hydrogen chloride is evolved. The product is poured into water, treated with sodium carbonate, and extracted with ether. It forms white prisms, of a silky lustre, insoluble in water, readily soluble in dilute acids, alcohol, ether, and benzene. It melts at 100° and distils at 295—302° without decomposition.

Para-γ-dimethoxyquinaldine, $C_9NH_4Me(OMe)_2$, obtained by heating the chloro-compound with sodium methoxide and methyl alcohol for two hours at 130—140° crystallises in needles or prisms melting at 94°; it is readily soluble in ether, alcohol, and in hot benzene, insoluble in water.

When the sodium compound of paramethoxy-γ-hydroxyquinaldine is heated with methyl iodide and benzene at 144—160° for three hours, and the methyl iodide derivative so obtained is digested with freshly precipitated silver oxide, *paramethoxymethylquinaldine*, $C_{12}H_{13}NO_2$, corresponding with methylquinaldine (Abstr., 1887, 680), is formed.

This crystallises in slender, lustrous needles, melts at 149°, and dissolves readily in boiling ethyl acetate, benzene, and alcohol, sparingly in ether; hot water dissolves it in all proportions.

Paramethoxy-γ-hydroxyquinaldine methochloride, $C_{11}H_{11}NO_2,MeCl$, crystallises in long, lustrous, white needles melting at 251°. The *platinochloride*, $(C_{12}H_{14}NO_2)_2,PtCl_6 + 4H_2O$, forms yellow needles.

Orthomethoxy-γ-hydroxyquinaldine, $C_{11}H_{11}NO_2 + H_2O$, is prepared in a manner exactly similar to the para-compound; dianisylcarbamide, melting at 182° (not 174°, Mühlhäuser, Abstr., 1882, 302), is formed in the reaction. It crystallises in long, matted needles of a silky lustre; the anhydrous compound melts at 229°. The *platinochloride* crystallises from chloroform in lustrous, reddish-yellow plates which melt with decomposition at 239°. N. H. M.

Aromatic Halogen Acetamido-compounds and their Derivatives. By P. W. Abenius (*Ber.*, **21**, 1665—1668).—*Diphenyldiketopiperazine*, $NPh<\begin{matrix}CO\cdot CH_2\\CH_2\cdot CO\end{matrix}>NPh$, crystallising in slender, white needles which melt at 263°, is obtained from brom- or chlor-acetanilide by the method already described (this vol., p. 824), or by heating chloracetylphenylglycine, $CH_2Cl\cdot CO\cdot NPh\cdot CH_2\cdot COOH$, with aniline at 140—150°. *Chloroacetylphenylglycine* crystallises in plates melting at 132—133°.

Dinitrodiphenyldiketopiperazine,

$$NO_2\cdot C_6H_4\cdot N<\begin{matrix}CO\cdot CH_2\\CH_2\cdot CO\end{matrix}>N\cdot C_6H_4\cdot NO_2,$$

is very sparingly soluble, and melts at a temperature too high to be accurately determined. The *amido-compound* crystallises in small, rhombic plates. When diphenyldiketopiperazine is boiled with alcoholic potash or with fuming hydrochloric acid, *phenylglycinylphenylglycine*, $NHPh\cdot CH_2\cdot CO\cdot NPh\cdot CH_2\cdot COOH$, melting at 129—130°, is formed. Phosphorous pentachloride acts on the piperazine-derivative with formation of the compound $\begin{matrix}CO\cdot CCl\cdot NPh\\|\quad|\quad|\\NPh\cdot CCl\cdot CO\end{matrix}$. This crystallises from alcohol with 1 mol. EtHO, in prisms melting at 247°.

Diparatolyldiketopiperazine, $C_6H_4Me\cdot N<\begin{matrix}CO\cdot CH_2\\CH_2\cdot CO\end{matrix}>N\cdot C_6H_4Me$ [N : Me = 1 : 4], is obtained together with *ethylglycolparatoluide*,

$$C_7H_7\cdot NH\cdot CO\cdot CH_2\cdot OEt,$$

which crystallises in large, lustrous prisms melting at 32°, by the action of alcohol on bromacetoparatoluide (m. p. 164°). It is also formed by heating paratolylglycine at 200°. It crystallises in slender prisms melting at 252—253°.

Bromacetoparaxylidine, $C_6H_3Me_2\cdot NH\cdot CO\cdot CH_2Br$, crystallises in needles melting at 145°.

Diparaxylyldiketopiperazine, $C_6H_3Me_2\cdot N<\begin{matrix}CO\cdot CH_2\\CH_2\cdot CO\end{matrix}>N\cdot C_6H_3Me_2$ [N : Me_2 = 3 : 1 : 4], melts at 203°.

Ethyl metamidocumate, $NH_2\cdot C_6H_3Pr\cdot COOEt$ [= 3 : 4 : 1], melts at

51—52°. The *bromacetyl-derivative*, $CH_2Br{\cdot}CO{\cdot}NH{\cdot}C_6H_3Pr{\cdot}COOEt$, melts at 106—107°. When the latter is boiled with alcoholic potash, the *acid* $COOH{\cdot}C_6H_3Pr{\cdot}N<\begin{matrix}CO{\cdot}CH_2\\CH_2{\cdot}CO\end{matrix}>N{\cdot}C_6H_3Pr{\cdot}COOH$ is formed; this does not melt when heated: the *ethyl salt* melts at 187—188°.

N. H. M.

Morphine Hydriodide. By H. Kunz (*Arch. Pharm.* [3], **26**, 307—311).—A concentrated alcoholic solution of potassium iodide mixed with a concentrated solution of morphine hydrochloride quickly becomes a compact, crystalline mass of morphine hydriodide in hair-like needles which only slowly dissolve on the addition of more spirit. The compound was found to be $C_{17}H_{19}NO_3,HI + H_2O$. Morphine hydrobromide can be similarly produced. J. T.

Atropine and Hyoscyamine. By W. Will (*Ber.*, **21**, 1717—1726).—Hyoscyamine forms a series of crystalline salts. The *sulphate* $(C_{17}H_{23}NO_3)_2,H_2SO_4$, crystallises from alcohol in slender, colourless needles melting at 206°. The *aurochloride* melts at 162°. The *hydrobromide* crystallises from water in compact prisms. The specific power of the base was found to be $[\alpha]_D = -20{\cdot}97$. Hyoscyamine is converted into atropine when it is heated for five hours at 109—110°, or when a trace of soda is added to an alcoholic solution, and the whole allowed to remain for some time. Ammonia acts similarly, but only very slowly. F. S. K.

Wrightine (Conessine) and Oxywrightine. By H. Warnecke (*Arch. Pharm.* [3], **26**, 248—261, 281—292; compare Abstr., 1886, 372).—Powdered seeds of *Wrightia antidysenterica* are freed from fat by means of ether; the powder is then digested with alcohol and hydrochloric acid at 60°, freed from alcohol by distillation and digestion with water, and concentrated ammonia is added in large excess. The precipitate is washed, pressed, rubbed up with sand, and extracted with light petroleum. The almost colourless solution is shaken up with solid potash to remove all water and allowed to evaporate; and the crystalline residue is purified by recrystallisation. The formula of wrightine is probably $C_{24}H_{40}N_2$; its platinochloride, hydrochloride, nitrate, and oxalate are described; its reactions in strong hydrochloric acid solution with alkaloïd reagents are also described in detail.

Oxywrightine is prepared by dissolving wrightine in 5 per cent. sulphuric acid and mixing this with dilute potassium iodate solution. After remaining 24 hours in the dark, the separated iodine is removed by chloroform and the colourless liquid is carefully treated with ammonia; after a short time a crystalline precipitate begins to form, and precipitation is rapidly completed on the addition of concentrated ammonia. The crystals of oxywrightine are anhydrous, colourless, compact needles of strongly alkaline reaction and bitter taste. This alkaloïd, like atropine and hyoscyamine, strongly reddens phenolphthaleïn, whilst wrightine is much less effective, and the other alkaloïds have still less action. Oxywrightine has the composition $4C_{12}H_{21}NO,C_{12}H_{19}NO_2$.

There are also described oxywrightine platinochloride, methyl hydroxide, methochloride, and methyl hydroxide and platinochloride.

The colourless solution of oxywrightine in eight drops of concentrated sulphuric acid when heated to 90—100° becomes yellow, passing gradually to violet-red; with two drops of sulphuric acid the yellow becomes an intense rose colour. This is characteristic, and clearly shows $\frac{1}{1000}$ mgrm. J. T.

Alkaloïds of Scopolia Japonica. By E. Schmidt and H. Henschke (*Arch. Pharm.* [3], **26**, 185—203).—10 kilos. of scopolia root was extracted with 90 per cent. alcohol; the filtered extract, when freed from alcohol and concentrated, deposited a granulo-crystalline fatty compound whose nature was investigated by Henschke. This substance was removed and the liquid was mixed with excess of potassium carbonate solution, when a bluish phosphorescence and odour of trimethylamine made their appearance. The alkaloïds were now removed by means of chloroform, the latter distilled off at the lowest possible temperature with the aid of water, acidified with sulphuric acid, and potassium carbonate in very light excess was added to precipitate the former. Further treatment with chloroform was necessary to remove potassium sulphate and purify the alkaloïds, but it was found impossible to obtain them in crystalline form, even on treatment with ether; a brownish syrup only resulted. To evaporate the alkaloïds, this syrup, dissolved in hydrochloric acid, was fractionally precipitated by gold chloride as suggested by Ladenburg. After repeated recrystallisation, hyoscine aurochloride, melting at 198—200°; hyoscyamine aurochloride, melting at 159—160°, and atropine aurochloride, melting at 136—138°, were isolated. J. T.

Alkaloïds of Scopolia Hlardnackiana. By E. Schmidt (*Arch. Pharm.* [3], **26**, 214—215).—The freshly gathered root of this plant was examined for alkaloïds by the method employed for *Scopolia japonica*. Hyoscyamine aurochloride was the only salt obtained. The alcoholic extract showed a blue fluorescence like that obtained from japonica, but there was not sufficient material available to ascertain whether this was due to the presence of scopoletin. J. T.

Application of Dialysis to the Study of the Gelatinous State of Albuminoïd Substances. By A. Solovieff (*J. Russ. Chem. Soc.*, 1887, 393—397).—Solutions of albuminoïd substances, after removal of the gelatinous coagulum, were subjected to the dialytic action of reagents by which they are gelatinised, and in this way the observations of the time of reaction as regards the penetration of the reagents (acids or bases) into the solutions of albumin, separated from them by a membrane, could be substituted for the measurement of the weight or volume of the reagents necessary to produce the same action. From white of eggs, gelatinous coagula are obtained by the action of organic acids or mineral bases, and the action is shown to be dependent on the strength both of the reagents and of the solutions acted on. The resistance which solutions offer to the conversion of albumin into the gelatinous form may be overcome even after the

action of the reagent has taken place through the membrane, by the action of substances which are capable of withdrawing water, such as alcohols, salts, &c. The conditions of their conversion into the gelatinous state are not dependent on any deep-seated change in the structure of the albumins. For example, if albumin is boiled with hydrochloric acid of 0·2 per cent. no coagulation takes place, although after concentration it will give gelatinous coagula, whereas the same solution after the reagent has been separated by dialysis is coagulated by boiling. The method of dialysis has the advantage that not only in the case of the action of organic acids, but also in that of strong bases, the albumin is gelatinised if the dialysis is stopped at the right time. The remaining part of the paper treats on the different states of albumin (especially Tarchanoff's "tata-albumin") and their mutual conversions and relations. B. B.

Silk. By T. Weyl (*Ber.*, **21**, 1407—1410).—The author finds that when commercial discharged silk completely deprived of silk-glue by digestion with 5 per cent. aqueous soda, is washed with 5 per cent. hydrochloric acid and finally with water (compare Staedler, *Annalen*, **111**, 12), and dried at 100°, it dissolves readily in cold fuming hydrochloric acid (sp. gr. = 1·2), and that the filtered solution when poured into a large quantity of cold 90 per cent. alcohol (400 c.c. to 5 grams of silk employed) yields a very voluminous, flocculent precipitate which rapidly forms a transparent, gelatinous mass. This compound, *sericoïn*, is obtained as a white powder by washing the precipitate with alcohol, boiling it first with absolute alcohol to completely dry it, then with dry ether, and finally allowing it to remain over sulphuric acid in a vacuum. So far as the percentage of carbon and hydrogen is concerned, sericoïn does not differ appreciably in composition from fibroïn, but it contains about 2 per cent. less of nitrogen. Sericoïn, even in very concentrated solution, is optically inactive, and when boiled for some time with dilute sulphuric acid yields, like fibroïn, a substance which gives the biuret reaction with copper sulphate and dilute aqueous soda. Fuchsin and picric acid colour sericoïn very slightly, but Millon's reagent gives with it a red colour as with fibroïn. Inasmuch as fibroïn is nothing more than discharged silk there is nothing to show that it has yet been obtained pure, and therefore sericoïn may be simply purified fibroïn; ammonium chloride has, however, been detected in the acid filtrate from sericoïn, and this would seem to point to the elimination of some of the nitrogen in fibroïn by the action of hydrochloric acid. W. P. W.

Silk. By T. Weyl (*Ber.*, **21**, 1529—1532; compare preceding Abstract).—When white silk, purified by Städeler's method, is boiled for 18 hours with sulphuric acid (1 : 5), the biuret reaction, which is clearly shown during the first 10 hours, disappears, and the whole dissolves to a yellowish-brown liquid on the surface of which a small quantity of a fatty acid swims in the form of oily drops. Tyrosine (5·2 per cent.) crystallises from the solution after removing the sulphuric acid by means of baryta-water. The filtrate from the tyrosine yields on evaporation a crystalline mass (15 per cent.) which

is most probably α-alanine. This compound crystallises in large, seemingly rhombic prisms, is tolerably readily soluble in cold water, ammonia, and soda, but is only very sparingly soluble in 40 per cent. alcohol and insoluble in ether. An aqueous solution is not precipitated by mercuric nitrate, and does not evolve ammonia when boiled with soda. The aqueous solution is optically inactive. When heated carefully, it sublimes in woolly flocks, and on further heating an oily, basic-smelling substance separates. When heated in a capillary tube, it remains unchanged at 230°, turns brown at about 237°, and at about 255° is converted into a brown, partly oily mass. Synthetical α-alanine has the same crystalline form, and behaves similarly when heated.

Glycocine (7·5 per cent.) was isolated from the mother-liquor from the alanine. F. S. K.

Alkali and Acid Albumin-derivatives. By M. Savin (*J. Russ. Chem. Soc.*, 1887, 398—402).—The following are the results of the author's investigation. The so-called ammonia-albuminate of white of eggs, and the acid albumin obtained by the action of hydrochloric acid of 0·2 per cent. after precipitation with ammonium sulphate and subsequent dialysis until all salt is removed and the reaction is neutral, are insoluble in water but soluble in acids and alkalis. The solubility in salt solutions depends on the time which has elapsed since their preparation. When the dilute albumin solutions are made alkaline and then digested on the water-bath with ammonia or with soda, the albumin is converted into globulin, but the conversion is incomplete. As regards their behaviour towards water, solvents, and reagents, the acid albumins are identical with the feebly alkaline derivatives of albumin (globulins) when in the form of pure precipitates. The same holds good with regard to ferments. The fact that globulins are obtained by "tryptonisation" seems to confirm the identity of globulins with albumins. B. B.

Spectrum of Acid Methæmoglobin. By H. Bertin-Sans (*Compt. rend.*, **106**, 1243—1245).—Considerable uncertainty exists as to the number of bands in the spectrum of acid methæmoglobin, some observers holding that the two bands between E and D are due to the presence of oxyhæmoglobin. The author finds that these bands are always present whatever the proportion of reagent used to convert the oxyhæmoglobin into methæmoglobin, and they only disappear when the solution is largely diluted. Moreover, the two bands are different in character and position from the bands of hæmoglobin. If potassium ferricyanide is added to a solution of carboxyhæmoglobin the spectrum of the solution is identical with that of methæmoglobin, and if the liquid is now treated with ammonia and ammonium sulphide the spectrum of carboxyhæmoglobin reappears.

The spectrum of acid methæmoglobin consists of four bands, the mean wave-lengths being 6330, 5800, 5385, and 5000 respectively. The first is the most intense; the second is about the same breadth, but is very feeble; the third is more intense than the second, and is about twice as broad; the fourth is seen only in a dilute solution, and is broader and more intense than either the second or third. This

spectrum has a general resemblance to the spectrum of hæmatin in an acidified alcoholic solution, but is more distinct. C. H. B.

Physiological Chemistry.

Digestion of Flesh in Normal Stomachs. By A. CAHN and v. MERING (*Bied. Centr.*, 1888, 352—353).—The acidity of the stomach juices is due solely to hydrochloric acid, which is present to the amount of 0·099—0·186 per cent. per hour (Human). In medium sized dogs, the quantity varies from 0·10—0·13 per cent. The percentage may rise after three hours to 0·39, and peptone may be present to the extent of 2·2 per cent. in one hour and 2·86 per cent. after two hours. After three hours, but little undissolved flesh meal can be found; consequently the author considers that the greater part of the albuminoïds are peptonised in the stomach of a healthy animal before they pass to the duodenum. In spite of the variation in acidity, the total quantity of hydrochloric acid remains the same. Syntonin, although in small quantities, is found to the very end of the process, and the quantities of peptones rise in proportion to the acidity.

E. W. P.

Influence exerted by Sodium Chloride on the Digestion of Albumin in Fodders. By SIEVERT (*Bied. Centr.*, 1888, 315).—The results obtained are given in the following table:—

	Per cent. albumin.	Coefficient of digestion.			
		Without NaCl.	With 0·5 per cent. NaCl.	With 1 per cent. NaCl.	With 2 per cent. NaCl.
Coarse bran	13·82	84·51	87·30	83·86	78·28
Coarse bran previously deprived of fat.....	15·70	77·96	75·74	78·53	78·28
Fine bran	19·25	88·00	88·00	90·44	90·23
Rye bran	12·95	89·96	87·88	87·88	87·88
Rice meal (1).....	13·47	87·36	87·00	88·90	—
„ (2).....	15·31	86·02	86·02	86·02	86·02
Palm-nut meal	16·54	75·90	—	75·40	—
Rape cake	30·89	85·85	85·85	86·80	86·90
Hemp cake.....	28·26	83·30	82·83	82·83	82·83
Cotton-seed cake	45·85	89·70	89·60	89·14	89·50
Earth-nut cake	45·15	94·35	94·00	93·80	94·30
Flesh-meal	69·32	94·30	—	94·20	94·20
Aftermath	13·40	71·50	71·80	72·91	72·91
Dried grains	21·61	74·30	73·73	74·30	74·30
Lupines	30·48	99·16	—	—	—
Lupines deprived of bitter principle	31·23	97·20	—	—	—

Stutzer's method was employed. E. W. P.

Microchemical Observations on Hyaline Cartilage. By C. T. Mörner (*Zeit. physiol. Chem.*, **12**, 396—404).—The cartilage investigated was that of the trachea. Morochowetz, Landwehr, and Krukenberg have obtained gelatin from such cartilage, and a second substance, the investigation of which was the object of the present research. The existence of two substances in the ground substance can be demonstrated by the use of certain staining reagents. Methyl-violet, indigo-red, and a mixture of ferric chloride and potassium ferrocyanide stain a trabecular network which pervades the ground substance and is continuous with the perichondrium. This is composed of a collagenous material, from which gelatin is obtainable. The other substance occurs in spherical masses (chondrin balls), which surround the cells, and correspond in position with the cell capsules. This is not stained at all by the reagents previously mentioned, but is stained by indigo-blue and tropæolin, which do not affect the collagenous network. By the use of two staining reagents, one of each group, and applied to microscopic sections, double staining is obtained: for instance, with tropæolin and methyl-violet the network is stained yellow, the chondrin balls blue; with indigo-blue and aniline-red the network is stained blue, the chondrin balls red. It is the chondrin balls which have evidently been the second substance of other authors, the mucin of Morochowetz and v. Mering, the hyalogen of Krukenberg.

The chemical investigation of the chondrin balls shows them to consist of two substances, each differing from collagen and also from one another. The chemical separation of the constituents of the cartilage is accomplished by the use of dilute hydrochloric acid, and subsequently of dilute potassium hydroxide solutions. The former only diminishes the transparency of the sections; the latter dissolves out the chondrin balls. The chondrin is precipitated from its alkaline solution by acids, and also by certain metallic salts. Further details concerning the chemistry of these substances are promised. The present paper is concerned with histochemical details only.

W. D. H.

Lactic Acid of the Thymus and Thyroid. By R. Moscatelli (*Zeit. physiol. Chem.*, **12**, 416—418).—Gorup-Besanez (*Annalen*, **98**, 1) showed that the thymus and thyroid contain lactic acid, but which form of lactic acid has not yet been settled. Hoppe-Seyler's method (*Handbuch*, 109) of isolating the acid was employed with slight modifications. The water of crystallisation and the amount of zinc in the lactate of zinc prepared from both sources showed that the acid in question is paralactic acid. W. D. H.

Production of Lactic Acid during Artificial Circulation of Blood through the Liver. By Wissokowitsch (*Chem. Centr.*, 1888, 117—118, from *Arch. Physiol.*, **87**, 91).—Experiments were made to determine what change occurred in the amount of lactic acid in the blood during artificial circulation through the liver. Arterial and venous blood obtained from a suffocated animal, and serum were used. In all cases, with one exception, in the case of venous blood, the lactic acid was considerably increased, not infrequently to double the original

quantity present in the blood. Liver is more active in the production of lactic acid than either muscle, kidney, or lung. Neither the amount of oxygen nor the presence of blood corpuscles are necessary for the increased production of lactic acid. From the positive result obtained with serum, it was possible that the increase of lactic acid was due to a simple washing out of the liver, but an experiment with 0·7 per cent. sodium chloride solution showed that this was not the case. J. P. L.

Nitrogenous Constituents of the Contents of the Intestine which arise from the Body itself and not from Food. By V. Hofmeister (*Chem. Centr.*, 1888, 116, from *Arch. Thierheilkunde*, **14**, 39—54).—To assume that the difference between the total proteïds ingested and the proteïds formed in the fæces strictly represents the proteïds digested would be an error, for the fæces contain, besides the undigested food material, the proteïds of the digestive fluids, epithelium of the digestive tract, mucus, and the nitrogen of the unabsorbed bile constituents. In order to estimate the limits of this error, the author has made a series of feeding experiments with pigs and horses on a purely non-nitrogenous (starch-paste) diet.

The amount of nitrogen expressed in terms of proteïd which arises from the above-mentioned causes in different parts of the alimentary canal varied during this diet very considerably. In the stomach, it varied from 0·17 to 14 per cent.; in the small intestine from 0·28 to 1 per cent.; and in the cæcum it was 0·65 per cent. The author explains how to apply corrections, and takes the opportunity to point out that during this starch diet the reaction of the cæcum contents was acid, whereas with other diets it was without exception alkaline. In the small intestine of the horse, too, the anterior portions were acid, whereas on other diets it was alkaline throughout. Sugar was almost completely absent from the stomach and small intestine, but present in the cæcum and even in the colon. This anomaly is explained from the want of amylolytic ferment in the food and from the very small secretion of saliva with this diet. In the cæcum, where the food remains some time, and where it meets with amylolytic ferments in its own secretions, sugar is formed, and it is to the formation of lactic acid from this sugar that the acid reaction of the cæcum is ascribed. J. P. L.

Variations in the Fat of Milk. By L. F. Nilson (*Bied. Centr.*, 1888, 171—183).—The author examined the milk of a herd of 15 cows in Sweden for the space of a year, making separate determinations in the case of each individual cow and of the morning's and evening's milk. These determinations were made every sixth day, and in some cases more frequently, and consisted in the estimation of the fat by Soxhlet's areometric method and of the volatile acids in the fat by a method giving relative values. He found, with but few exceptions, that the evening's milk was richer in fat than the morning's milk. The former gave 3·82 per cent. of fat and the latter 3·33 per cent. as an average of the whole year. As regards volatile acids, the fat of colostrum contained an unusually small amount; the

amount then rose rapidly, reaching a maximum in from five to seven days from calving; at this height, it remained for a short time, and then slowly fell until the end of the period of lactation.

The difference between summer and winter butter, depending on the amount of low melting glycerides, is usually ascribed to pasturing and fodder, but the author believes it to be due rather to the fact that the cows are usually in the pastures and receiving green fodder at the earlier part of their period of lactation.

The author found that the volatile acids were larger in amount in the fat of morning's than of evening's milk from November to March, that in April the difference disappeared, that during the spring and summer it was very small, and that it reappeared in the autumn. This he ascribes to the darkness of the Swedish autumn and winter nights, and the lightness of those of the spring and summer.

H. H. R.

Changes in the Composition of Milk. By H. Faber (*Bied. Centr.*, 1888, 316—317).—Examination of very numerous milk analyses shows that the dry matter minus fat remains constant (8·7 to 8·8), but that the fat alone varies. Evening milk contains most fat, as also does that produced in October and November. E. W. P.

General Method of obtaining Non-organised Ferments in pure Aqueous Infusions. By N. Kravkoff (*J. Russ. Chem. Soc.*, 1887, 387—392).—The non-organised ferments, together with albuminoïds, are precipitated by ammonium sulphate. By treatment with alcohol, the albuminoïds become insoluble, and the ferments can then be extracted with water. From human saliva, the ferment was obtained as follows: the saliva was mixed with an equal volume of water, and finely-powdered ammonium sulphate was added to complete saturation, leaving some of the salt undissolved. After filtration, the precipitate was boiled for five minutes with strong alcohol, in order to render it more compact and easier to remove from the filter; it was then placed in absolute alcohol from 1 to 1½ days. After carefully decanting the alcohol, the precipitate was dried at 30°, and then extracted with a volume of water equal to that of the original saliva. On filtration, a clear liquid was obtained, showing but a slight opalescence, and quite free from albuminoïd or mucous substances. At 40°, a turbid starch solution is rendered clear at once. Fehling's test is reduced. This ferment infusion loses its activity in the air, whereas in an atmosphere of carbonic anhydride or chlorine, its hydrolytic property remains unaltered. In the same way, an infusion of trypsin (better pancreatin) was obtained from the salivary gland of a dog's stomach, or the stomach of a calf or pig; it was found that the pancreas infusion obtained from the dog had the most energetic action, that from the calf being the most feeble. The filtrates from the quasi-mechanical precipitation with ammonium sulphate contain no active ferment, and the above pure ferment infusion, on addition of ammonium sulphate, becomes turbid, and the flocks which separate, after being treated in the same manner as the precipitates obtained from saliva, give extracts with the same

hydrolytic (saccharifying) properties as the above infusions, whilst the filtrates are found to be inactive. B. B.

Active Oxygen in Living Tissue. By C. WURSTER (*Ber.*, **21**, 1525—1528).—A reply to Bokorny (this vol., p. 751), in which the author gives further evidence in support of his views. Many vegetable juices oxidise more strongly than a centinormal iodine solution. The saliva of healthy, fair, young men quickly oxidises ammonia in glacial acetic acid solution to nitrous acid. For reasons previously stated the oxidising substance in vegetable juices must be either nitrous acid or hydrogen dioxide, and although hundreds of juices show this strong oxidising action, nitrous acid could not be detected, by means of Griess' reagents, in a single case. F. S. K.

Furfuraldehyde Reactions. By L. v. UDRÁNSZKY (*Zeit. physiol. Chem.*, **12**, 377—395; compare this vol., pp. 180 and 878).—The question whether normal urine contains carbohydrates has been one on which a large amount of work has been done, but has never been satisfactorily settled. The most recent of these observations are those of Landwehr (Abstr., 1887, 26, this vol., p. 175), who states that animal gum may be present in the urine, and the author's own researches on humous substances (this vol., p. 180). The fact that carbohydrates yield furfuraldehyde on treatment with acid, which can be identified by means of the characteristic colour given with bile acid, α-naphthol, and many other substances, has led to the present reinvestigation of the question. The special method adopted is that of H. Schiff (*Ber.*, **20**, 540), in which strips of filter-paper are dipped in a mixture of xylydine, glacial acetic acid, and alcohol, then dried. The substance suspected to contain carbohydrate is heated in a tube with sulphuric acid; the fumes, which contain furfuraldehyde, strike a red colour with the strips of paper placed at the mouth of the tube. Using this method with quite small quantities (a few drops) of normal urine, the colour never fails to appear. The conclusion is drawn that normal urine contains carbohydrates, although of what kind is doubtful. A reaction described by Molisch, in which either thymol or α-naphthol and sulphuric acid give a red coloration, may also be used with the same result.

In cases of glycosuria these reactions occur more readily, and by a minutely described process of appropriate dilution of the urine, an approximate quantitative result may be obtained, at least sufficiently near to enable one to say whether the secretion contains more than a normal amount of carbohydrate.

In all such testing, the urine must be free from proteïd, as the concluding portions of the paper show that furfuraldehyde is one decomposition product of proteïd; this fact is considered to be the first well-established chemical relationship between proteïds and carbohydrates, although the physiological connection between the two classes of substances has been long recognised (Bernard, Seegen, &c.). It also affords an explanation of the colour reactions which are caused by treating proteïd with acid, such, for example, as the Adam-

kiewicz reaction. The amido-acids which result from the decomposition of proteïds yield no furfuraldehyde. W. D. H.

Pathological Pigments. By E. Neumann (*Chem. Centr.*, 1888, 227—228, from *Virch. Arch.*, **111**, 25—47).—(1.) *Origin of Hæmatoidin and Hæmosiderin in Extravasations and Thrombi.*—All observations lead to the conclusion that hæmosiderin arises only from those blood corpuscles which are in intimate contact with, or penetrate into the tissue, and that hæmatoidin arises from blood corpuscles which remain in the coagulum outside the tissue; for the formation of hæmosiderin, the action of the living tissue or its cells is necessary, whilst the formation of hæmatoidin is not conditional on the vital activity of tissue, but on chemical decomposition. (2.) *Pseudomelanose.*—The black pigment grains of pseudomelanose are, like hæmosiderin granules found almost exclusively within the cellular elements.

The change of iron pigments from rusty-brown to black when putrefaction sets in, favours the assumption that pseudomelanotic pigmentation of tissue is brought about by a combination of pathological and cadaveric processes. J. P. L.

Physiological Action of Cocaïne. By Mosso (*Arch. Pharm.* [3], **26**, 179, from *Ann. Chim. Farm.*, 1887, 326).—The author finds experimentally that cocaïne is the best cerebro-spinal stimulant. If an animal sleeping from a fatal dose of chloral hydrate be injected with 0·01 to 0·02 gram of cocaïne per kilo. of body-weight, the rate of breathing and pulsation is increased, and the animal awakes, although the low temperature induced by the chloral hydrate continues. Conversely, the action of 0·046 gram of cocaïne per kilo. of the body-weight is neutralised by 1·5 grams of chloral hydrate; cocaïne may therefore prove to be efficacious in the case of poisoning by means of opium, morphine, and other narcotic substances, which strongly depress the nerve-centres governing respiration and the heart's action. J. T.

Behaviour of Benzoic Anhydride in the Organism. By E. Salkowski (*Chem. Centr.*, 1887, 186, from *Centr. Med. Wiss.*, **25**, 161—163).—Benzoic anhydride shows the same marked antiseptic properties as the acid; when it is digested with finely minced meat or albumin at 40°, it is converted into the acid, but no sepsis occurs. Digestive ferments cause the same change. Taking benzoic anhydride does not induce any evident disturbance; it is changed into benzoic acid, and excreted in the urine as hippuric acid. The antiseptic action in the intestine is shown by the diminution in the amount of ethereal sulphates which occurs in the urine. Its action on the proteïd metabolism in dogs is small, and not greater than the action of benzoic acid or sodium benzoate. J. P. L.

Behaviour of Orthohydroxyquinolinecarboxylic Acid and its Derivatives in the Organism. By S. Królikowski and M. Nencki (*Monatsh.*, **9**, 208—216).—Orthohydroxyquinolinecarboxylic

acid has no injurious action on rabbits when administered subcutaneously in doses of 1 gram of the sodium salt. Daily doses of 2 to 4 grams were given to a dog weighing 12 kilos. without any injurious effect. The animal's urine was deep yellow, showed an acid reaction, and gave the same deep brownish-red coloration with ferric chloride as orthohydroxyquinolinecarboxylic acid. Part of the acid separated unchanged when the urine was evaporated, and a further quantity was obtained when the filtered urine was acidified with acetic acid, and treated with cold alcohol. The greater part of the acid had therefore passed unchanged, and no new product could be detected, neither was the formation of ethereal sulphates increased to any appreciable extent.

Methyltrihydro-orthohydroxyquinolinecarboxylic acid has no injurious effect on dogs in doses of 2 grams daily, nor has it any deleterious action on man when taken in doses of 1 gram from 4 to 5 times daily. The urine shows the same deep-red coloration with ferric chloride as the free acid; when it is evaporated, extracted with alcohol, and the alcoholic extract evaporated to a syrup, acidified with hydrochloric acid, and shaken with ether, a crystalline precipitate of unchanged methyltrihydrohydroxyquinolinecarboxylic acid (70 to 80 per cent.) separates after a time.

Methyldihydrodihydroxyquinolinecarboxylic acid,

$$C_9NH_5Me(OH)_2{\cdot}COOH,$$

is present in very small quantities in the ethereal extract. It crystallises in rhombic needles and prisms melting at 254—255°, is almost totally insoluble in water, but readily soluble in alcohol, ether, and alkalis, and gives a blue coloration with ferric chloride. Acids precipitate it from its solution in alkalis as a white, amorphous compound. This compound is formed to a somewhat larger extent in the human than in the canine organism.

The quantity of the ethereal sulphates in urine is not increased by doses of methyltrihydrohydroxyquinolinecarboxylic acid.

It seems that a small portion of tetrahydro-orthohydroxyquinolinecarboxylic acid is oxidised to the dihydrodihydroxy-acid in the animal organism. F. S. K.

Reduction of Oxyhæmoglobin in Typhoid Fever. By A. Hénocque and G. Baudouin (*Compt. rend.*, **106**, 1245—1248).—Periods of high temperature correspond with periods of diminished activity of reduction, and when the temperature is reduced, activity of reduction increases and tends to regain its normal value. A relapse, by producing an increase of temperature, immediately reduces the activity of reduction. C. H. B.

Chemistry of Vegetable Physiology and Agriculture.

Bacteria of Normal Milk Fæces. By A. Baginsky (*Zeit. physiol. Chem.*, **12**, 434—462).—These observations were undertaken in hope that they might throw some light on the disorders of the

alimentary canal of suckling children. Escherich (*Die Darmbacterien des Säuglings*, Stuttgart, 1886) has shown that in the normal fæces of such children there are many forms of micro-organisms, but he dealt especially with two, the *B. lactis aërogenes* and the *B. coli commune*. In the present research these were separated and cultivated by the best modern methods, and portions of the pure cultivation of *B. lactis* (with which this paper exclusively deals, and which is so called on account of its supposed action in forming lactic acid from milk-sugar) were added to portions of the substances used as food by children.

With milk-sugar, an acid reaction is always produced, and after 20 days' fermentation about 80 per cent. of the sugar has disappeared. During the first few days, there is abundant formation of gas (carbonic anhydride, hydrogen, and methane). The merest traces of lactic acid are found, and sometimes it is entirely absent. In the distillate, traces of acetone are present. The acid formed is acetic acid. The same change occurs in the absence of oxygen; addition of bile to the mixture causes no difference. Lactic acid is not an intermediate product, for the action of the bacterium on lactic acid is to transform it not into acetic but into butyric acid.

The chief action of the ferment on starch is also to form acetic acid, but only small quantities are formed in the absence of oxygen. No reducing substance is ever produced. The ferment has no action whatever on caseïn; the total proteïd in milk before and after fermentation is the same. There are no products of the fermentation of proteïds (leucine, &c.) to be found; and the acid reaction seems to be simply due to the formation of acetic acid from milk-sugar. The power of various antiseptics to stop the fermentative action of the bacterium are finally adverted to; among the facts mentioned, the most interesting is the power of calomel both to stop the activity of the ferment, and to cure certain forms of infantile diarrhœa. The name *B. aceticum* (instead of *B. lactis*) is suggested.

W. D. H.

Influence of Filters on the Constitution of Water. By A. J. C. Snyders (*Ber.*, **21**, 1683—1691).—The author's results are given in tabular form, showing the bacteriological and chemical difference between various samples of filtered and unfiltered water after various lengths of time.

Sea-sand removes the greater part of the micro-organisms, but not all; it likewise exercises some slight chemical action on the water.

Chamberland-Pasteur filters (compressed asbestos) remove all bacterial germs for at least two months, but their chemical action is quite inconsiderable. The Chamberland filter (animal charcoal) has, however, a not inconsiderable chemical action, inasmuch as it removes the solid constituents, organic substances, chalk, and magnesia; ammonia, the nitrites, iron, and lead are partially kept back, but the chlorides are not acted on.

Maignen's "watch-filter" (carbo-calcis) removes all bacteria at first, but its action does not continue as long as that of the Chamberland filter. On the other hand, it has a very important chemical purifying action; about two-thirds of the solids are removed, the quantity of

ammonia and nitrites greatly diminished, and the whole of the organic substances, iron, and lead eliminated. The chlorides are not acted on. F. S. K.

Proteolytic and other Ferments in Oats, and their Action on Digestive Organs. By ELLENBERGER and V. HOFMEISTER (*Bied. Centr.*, 1888, 319—321).—A mixture of oats and water develops three ferments, an amylolytic, a proteolytic, and a lactic, all of which are destroyed at 100°; the action of such ferments greatly aids digestion, whereas increased activity of the digestive organs is required if the food is previously boiled. The principal of the ferments is the amylolytic, 2 per cent. of sugar being produced in three hours, or 2 per cent. lactic acid and 3·2 per cent. sugar in seven hours. The lactic ferment is much feebler, the action in three hours being only 0·5 per cent., and in 6 to 7 hours 1 per cent. Ice-cold water retards the action. The result of giving boiled food generally to Graminivoræ is to produce catarrh of the stomach, &c., because by the destruction of the ferments unusual action of the digestive organs is necessitated. E. W. P.

Diastase. By J. FANKHAUSER (*Bied. Centr.*, 1888, 205—207).—During the germination of barley, the development of the plumule is accompanied by the destruction and solution of the cell walls in the grain. This solution may be due either to a substance formed by the embryo, namely diastase, or to micro-organisms; a microscopical examination, however, failed to show the presence of the latter.

Experiments showed that during germination both of potatoes and of barley, the evolution of carbonic anhydride is accompanied by the secretion of one or of several powerful acids. Malt treated with 5 per cent. solution of potash yielded an extract, which after suitable treatment gave a distillate, in which formic acid was the chief constituent. It was found that both this distillate and also commercial formic acid had the power of changing a carbohydrate into sugar.

The author believes that the cell walls of the starch granules which are in direct contact with the plumule are attacked by the formic acid it secretes, and that in the course of brewing, the formic acid acts on the starch just as dilute sulphuric acid would. He also attributes the sweetness that potatoes acquire to a similar cause; formic and probably other allied acids are formed during the respiratory process that accompanies sprouting, and these acids attack the cell walls and also change the starch into sugar. Many other phenomena of plant growth can be explained by this secretion of strong acids by organs containing no chlorophyll, for instance, the piercing of wood by the mycelia of fungi. H. H. R.

Function of the Colouring Matter of Chlorophyll. By A. HANSEN (*Bied. Centr.*, 1888, 357—358).—Like the colouring matter of blood, the absorption-bands of chlorophyll have no connection with the assimilative power of the same. The colouring matter absorbs carbonic anhydride, and forms with it an unstable compound, which passes on the carbonic anhydride to the plasma of the chlorophyll

grains. For the formation of 25 grams of starch about 20 litres of carbonic anhydride (50 cm. air) are requisite, this quantity must pass over a square meter of leaf surface in 15 hours. Chlorophyllous cells absorb more and more gas as the temperature rises, consequently the law of diffusion and absorption of gases is not followed in this case; but the absorption is dependent on atmospheric pressure. E. W. P

Formation of Organic Acids, Nitrogenous Compounds, and Potassium Nitrate in Beetroot. By H. Leplay (*Compt. rend.*, **106**, 1020—1022).—Sugar-beet was sown in Fontainebleau sand, which had previously been heated to redness to remove organic matter and then mixed with 1 per cent. of calcium phosphate and 1 per cent. of calcium sulphate. The crop was watered daily with a liquid which contained in each litre 0·1 gram of potassium hydrogen carbonate, 0·1 gram of ammonium hydrogen carbonate, 100 c.c. of a saturated solution of calcium sulphate, and 100 c.c. of a saturated solution of carbonic anhydride. The beet was sown on May 26th, and the crop gathered on October 14th. During growth, the plants and soil were freely exposed to the air, but were protected from rain. The quantity of each substance in the crop for every one part of the same substance in the seeds was as follows:—Total matter, moist, 74·7; water, 581·00; dry solid matter, 8·66; organic and nitric nitrogen, 10·32; nitric nitrogen in the leaves, 15·50; potassium combined with organic acids, 17·43; calcium combined with organic acids, 17·38.

No calcium, ammonium, or potassium was present either in the form of oxides, or as hydrogen carbonate, and hence the nitrogen which is absorbed as ammonia becomes converted into nitrogenous organic compounds, and the calcium and potassium absorbed in the form of hydrogen carbonates are transformed into salts of organic acids (compare Abstracts, 1886, 830). C. H. B.

Synthesis of Albumin in Chlorophyll-containing Plants. By Chrapowitzki (*Chem. Centr.*, 1888, 185, from *Bull. Acad. St. Petersb.*, **32**, 96—98).—Plants which consume their own reserve supply of albumin when deprived of a supply of nitrogen, were used to determine the position where the new formation occurred. After exposing them to light, in mineral solutions free from nitrogen, until the albumin had disappeared from the leaves, they were transferred to nutrient solutions containing nitrogen.

Three to six days after the transference from the non-nitrogenous to the nitrogenous soil, an accumulation of albumin was found in the chlorophyll grains. The author concludes that the chlorophyll substances not only synthesise carbohydrates, but albumin also.

J. P. L.

Some Nitrogenous Constituents of the Seedlings of Soja Hispida. By E. Schulze (*Zeit. physiol. Chem.*, **12**, 405—415).—These researches are a continuation of previous observations on the seedlings of *Lupinus* and pumpkins (Abstr., 1887, 369, 747, &c.). The method adopted for the separation of the various bases are variations on those of Brieger. The bases were purified by recrystallisation, their characteristic tests were tried, and finally they, or some

characteristic salts, were subjected to elementary analysis. The final results showed that many bases, the products of retrogressive metabolism, exist; these resemble in kind those of *Lupinus* rather than those obtained from pumpkin sprouts, the chief difference being the almost complete absence of arginine in the seedlings of soja. The etiolated lupin-seedlings yield asparagine in great abundance, amidovaleric acid, phenylamidopropionic acid, arginine, choline, substances of the xanthine-group, and doubtful traces of leucine and tyrosine. The pumpkin sprouts yield very little asparagine, much glutamine, leucine, tyrosine, arginine, choline, vernine, and substances belonging to the xanthine-group. Richardson and Crampton have shown the presence of allantoïn in young wheat stalks. The amount of asparagine yielded by the soja seedlings was 7 to 8 per cent. of the dry material.

W. D. H.

Distribution of Amygdalin and Emulsin in Almonds. By W. Johannsen (*Bied. Centr.*, 1888, 326).—Amygdalin is found only in the parenchyma cells of the cotyledons. Emulsin was estimated by determining the fermentative power of the several portions of the almond. It appears that there is present in bitter almonds an amount of emulsin sufficient to convert 40 times the quantity of amygdalin usually present. Besides being present in the root shoots, it is also found in the fibrovascular bundles of the cotyledons, whereas it is only absent from the parenchymatous tissue of the curved sides of the cotyledons. Emulsin is likewise found in a similar situation in sweet almonds. The emulsin does not act on the amygdalin, owing to the fact that they are not in contact, but in different parts of the almond.

E. W. P.

Substance containing Sulphur found in Cruciferous Plants. By J. Smith (*Zeit. physiol. Chem.*, **12**, 419—433).—With the exception of ethereal hydrogen sulphates, the substances in the animal body which contain sulphur are exclusively proteïd or derived from proteïd. In plants, on the other hand, numerous sulphur-containing substances are found, the direct relationship of which to proteïd is not yet proved. Moreover, these substances are limited to certain natural orders, among which are the Crucifers. Bussy (*Annalen*, **34**, 223) has described potassium myronate in black mustard. Will (*Chem. Centr.*, **148**) has described a glucoside, sinalbin, in white mustard seeds; other similar observations are quoted.

The amount of uncombined and of combined (as ethereal hydrogen sulphates) sulphuric acid was estimated in the seeds of 19 varieties of Crucifiers; the former occurs either not at all, or in mere traces, except in *Isatis tinctoria*, and here it is probably in the shell of the seed. The ethereal hydrogen sulphates were found abundantly in all, but especially in the seeds of *Sinapis nigra*. In this form of mustard seed, about one-third of the total sulphur is combined as proteïd, the remaining two-thirds as myronic acid. A ferment in the seeds splits up the glucosides or the ethereal hydrogen sulphates; this occurs at the summer temperature, when the powdered seeds are suspended in water. In some seeds (for instance *Brassica napus*), the process occurs slowly, and at the end of two days has hardly begun; in other cases

(for instance, *Sinapis alba* and *Lepidium sativum*), it is almost completed in the same time. The ferment action can be stopped at any time by the addition of 0·3 per cent. of hydrochloric acid. Simultaneously there is an increase in the amount of uncombined sulphuric acid. This is illustrated by tables of analyses. An exactly similar change occurs in the germination of the seeds. Further it is shown that the seeds of one species freed from ferment by boiling, can be acted on by mixture with the seeds of another species, but there is considerable variation in the activity of the ferments prepared from various species. W. D. H.

Influence of Manures on the Composition of Barley. By KRANDAUER (*Bied. Centr.*, 1888, 297—298).—"Chevalier," "Franken," and "Halletts" barley were manured with nitre and guano superphosphate, and with nitre with half as much again of superphosphate. Only Franken barley answered to the smaller admixture of superphosphate as regards the grain, but an increase of straw was obtained in all cases. No decided increase of nitrogenous matter was induced. E. W. P.

Effect of Chemical Manures on the Composition of Soja. By A. LEVALLOIS (*Compt. rend.*, **106**, 1014—1017).—The soil employed in these experiments contained per kilo., 0·424 gram nitrogen, 0·480 gram potassium oxide, and 0·639 gram phosphoric anhydride. Two plots were planted with soja, and one of them was treated with a complete chemical manure. The manured plot gave a much heavier crop, but the proportion of grain was smaller, and its quality inferior. In the manured crop, the weight of the grain was to the total weight of the plant as 1 : 3·9, whilst with the unmanured crop, the ratio was 1 : 1·9. Moreover, in the latter case the grains were larger and more regular. The following table gives the proportion of various constituents in 100 grams of the dry grain in each case:—

	N.	P_2O_5.	K_2O.	Oil.	Substances soluble in alcohol.
Manured....	6·16	1·25	3·03	19·06	22·20
Unmanured..	6·27	1·47	3·16	20·33	19 60

The differences are probably due to the fact than in the first case the grains are smaller, and hence there is a greater proportion of testa to a given weight of grain. C. H. B.

Relation between Atmospheric Nitrogen and Vegetable Soils. By T. SCHLOESING (*Compt. rend.*, **106**, 982—987, 1123—1129).—The author has repeated his former experiments (this vol., p. 747), but has modified the method of manipulation in order to avoid the presence of mercury vapour, which is injurious to certain lower organisms. The soils employed were those of Grenelle and Montretout, which are comparatively poor in organic matter, and the results agree with those previously obtained; that is to say, that whilst there is a decided absorption of oxygen, the quantity of nitrogen removed from the atmosphere is too small to be measured by eudiometric methods.

The second paper is a reply to Berthelot (next Abstract). The author points out that Boussingault recognised that soils contain living organisms which exert an important influence on the formation and destruction of ammonia and nitrates. The conditions in his own experiments were not materially different from those in which Berthelot and André obtained evidence of the direct absorption of atmospheric nitrogen. The soils employed contained from 7 to 19 per cent. of clay, which is a considerable proportion, and the methods of eudiometric analysis are at least as accurate as determinations by means of soda-lime. C. H. B.

Absorption of Nitrogen by Soils. By BERTHELOT (*Compt. rend.*, **106**, 1049—1055).—A criticism of Schloesing's papers (this vol., p. 747 and preceding Abstract). The conditions in Schloesing's experiments were not similar to those in which the author and André proved the direct absorption of atmospheric nitrogen, and the analytical results cannot be accepted as accurate, because of the difficulties of manipulation inseparable from eudiometric methods, and also on account of the small quantity of soil employed. It is pointed out that Boussingault regarded the soil simply as a chemical supporter of vegetation, and had no regard to the minute living organisms which are always present. C. H. B.

Absorption of Nitrogen by Vegetable Soils. By BERTHELOT (*Compt. rend.*, **106**, 1214—1215).—A further reply to Schloesing.

Absorption of Nitrogen by Soils and Plants. By A. GAUTIER and R. DROUIN (*Compt. rend.*, **106**, 1098—1101, 1174—1176, 1232—1234).—The experiments were made with the artificial soils already described (this vol., p. 746). Determinations of nitric nitrogen always gave negative results, and hence it follows that nitrates are not the only source from which plants can obtain their nitrogen.

In all the experiments with soil free from organic matter, there was a loss of ammoniacal nitrogen, a gain of organic nitrogen, but a loss of total nitrogen. In the case of the soils containing organic matter, there was a gain of total nitrogen, and a gain in organic nitrogen, but a loss of ammoniacal nitrogen.

When the soil is supporting a crop, there is always a gain of total nitrogen and a gain of organic nitrogen, but a loss of ammonia. The gain in organic nitrogen is greatest with the soils which contain organic matter.

The loss of ammoniacal nitrogen in the case of the soils free from organic matter, is due to cryptogamic vegetation. A green coloration is observed on the surface of the soil, and this consists of unicellular algæ, such as *Pleurococcus vulgaris* and *Protococcus viridis*. They probably act, not by directly absorbing nitrogen from the air, but by converting into organic nitrogen the ammoniacal nitrogen.

The general conclusions arrived at from these and previous results (*loc. cit.*) are as follows :—Soil absorbs nitrogen from the air, provided that it contains organic matter, which is an indispensable agent in the process. Iron oxides accelerate the absorption, but are not essential.

Whatever may be the initial form of the nitrogen absorbed, it is converted into organic nitrogen. On the other hand, the soil continually loses the ammoniacal nitrogen brought to it by the wind and rain, or produced by bacterial fermentation in the soil itself. The permeability of the soil, its degree of subdivision and compression, are important factors, and if the soil is rammed down, or is rendered more or less impervious by the presence of excess of clay, very much less nitrogen is absorbed; hence the necessity in practice for a repeated breaking up and renewal of the surface. In the experiments described, the total quantity of nitrogen assimilated in three months in soil without plants is, for the area of surface exposed, 10 times as great as the ammonia absorbed by dilute acid in Schloesing's experiments. Since nitrates were absent it follows that there are in the air other substances besides ammonia and nitric acid from which nitrogen can be assimilated. The growth of plants on the soil results in more than double the increase in the quantity of nitrogen absorbed, and it follows that phanerogamic plants absorb, indirectly through the soil in which their roots are placed, or directly by their leaves, some of the free or combined nitrogen in the atmosphere, and fix it in their tissues.

Unicellular aerobic organisms, and especially certain species of algæ, which are universally distributed over the surface of arable soils, play an important part in the absorption of nitrogen by the soil, even when the latter is free from vegetable organic matter.

C. H. B.

Value of Nitrogen in Sodium Nitrate and in Ammonium Sulphate. By G. Klien (*Bied. Centr.*, 1888, 166—168).—Experiments were made on four different estates as to the comparative value of the nitrogen in these manures applied to oats and barley. In the results obtained, nitrogen in the one form appears sometimes better, sometimes of equal value, and sometimes inferior to nitrogen in the other form, so that no general conclusions can be drawn.

H. H. R.

Experiments with Farmyard Manure. By E. Heiden (*Bied. Centr.*, 1888, 154—165).—Strewing the manure in the stalls with superphosphate dried by gypsum, to the amount of 2 lbs. daily for every 1000 lbs. of live weight of the cattle, was found to cause a greater weight of manure, and a manure richer in nitrogen and phosphates than when this treatment was omitted. Further the manure thus treated lost less dry matter on keeping, and when used for potatoes gave a better yield than the untreated manure.

In the case of manure allowed to remain beneath the cattle for weeks, the effect of strewing with superphosphate dried by gypsum was found to be the production of a greater weight of manure, of dry manure substance, and also of manurial nitrogen.

Sulphuric acid containing phosphoric acid has recently been introduced in commerce as an agent for preventing the loss of combined nitrogen from liquid manure. The author experimented with a sample containing 3·72 per cent. of phosphoric acid, and 24·49 per cent. of sulphuric acid, adding it to the liquid manure in quantity sufficient to produce a slightly acid reaction, and repeating the addition several

times in order to secure this. He found that it saved a large proportion of the nitrogen that otherwise would have been lost.

H. H. R.

Prevention of the Loss of Nitrogen in Farmyard Manure. By B. C. DIETZELL (*Bied. Centr.*, 1888, 168—171).—Loss by volatilisation of ammonia and ammonium carbonate can be sufficiently prevented by treading the manure well down and keeping it moist, without employing kainite, gypsum, or ferrous sulphate to retain the ammonia. Loss by the formation of free nitrogen is not prevented by the addition of gypsum, calcium carbonate, or earth, and kainite only checks it by hindering decay, and the action ceases when the manure is applied to the land. The addition of sulphuric acid to liquid manure is not more effectual.

In some manuring experiments the author observed that no loss of nitrogen occurred in a soil manured with superphosphate. He explained the phenomenon by the supposition that calcium phosphate reacts with ammonium nitrite, and so prevents the action of nitrous acid on primary amines and amido-acids, and the consequent evolution of free nitrogen. Further experiments, in which precipitated phosphate and basic slag meal were used, confirmed his previous observation. He recommends that part of the phosphatic manure, instead of being put on the field, should be previously mixed with the farmyard manure, and in the case of liquid manure, that superphosphate should be placed in a basket and suspended in the liquid. H. H. R.

Analytical Chemistry.

Testing Mercury Oxides for Chlorides. By T. FELS (*Arch. Pharm.* [3], **26**, 29—30).—The silver nitrate test is much more sensitive when the oxide is dissolved in acetic acid than when either nitric or sulphuric acid is used as the solvent. J. T.

Detection of Copper in Wine. By GIGLI (*Arch. Pharm.* [3], **26**, 273—274, from *L'Orosi*, 1887, 397).—200 c.c. of wine is concentrated to one-fifth and strongly acidified with hydrochloric acid, when a small zinc-platinum couple is immersed. Any copper which may be present is deposited on the platinum plate within 24 hours. Iron alone causes no deposition on the platinum, but if copper is present, the two metals are deposited together, and require separation before the copper can be estimated quantitatively. J. T.

New Method for the Estimation of Manganese. By L. SCHNEIDER (*Monatsh.*, **9**, 242—254).—Bismuth tetroxide, in presence of a large quantity of nitric acid, oxidises manganous salts to permanganic acid quickly and completely at the ordinary temperature. When about 2 c.c. of nitric acid, sp. gr. 1·2, is present for every 1 mgram. of manganese, no precipitation of manganese peroxide occurs, and on

adding an excess of finely-divided bismuth tetroxide, oxidation is complete in a few minutes. The presence of considerable quantities of ferric oxide or sulphuric acid does not affect the results. This process can be employed for the estimation of manganese in iron, steel, &c., as follows:—5 grams of steel or iron, poor in manganese, is dissolved in 100 c c. of nitric acid, sp. gr. 1·2; when solution is complete, the whole is boiled for some time, allowed to cool, and about 100 c.c. of nitric acid, sp. gr. 1·2, added. The mixture is then digested for a few minutes with about 10 grams of bismuth tetroxide. In the case of iron which contains several per cent. of manganese, 1 gram is dissolved in nitric acid, and the solution diluted with 200 to 300 c.c. of the same acid. Ores or dross which are insoluble in nitric acid are boiled with hydrochloric acid, then treated with hydrofluoric acid and evaporated with sulphuric acid. In analyses of spiegeleisen rich in manganese, ferromanganese, and rich manganese ores, the solution containing 1 gram of the substance is diluted to 1 litre with water, 200 c.c. of this solution taken, mixed with 100 c.c. of concentrated nitric acid, and oxidised with bismuth tetroxide as before. When oxidation is complete, the solution of permanganic acid is filtered from the bismuth tetroxide through an asbestos filter, and titrated with a solution of hydrogen peroxide. This method can be carried out in about an hour, and the results are very satisfactory. The presence of free nitric acid is necessary during titration. The hydrogen peroxide employed for titrating the permanganic acid, is standardised by adding dilute nitric acid (1 vol. concentrated acid with 2 vols. of water) to a known quantity of potassium permanganate, until about 2 c.c. of nitric acid, sp. gr. 1·2, is present for every 1 mgram. of manganese, and then adding ordinary commercial hydrogen peroxide, diluted to about 0·5 per cent., until the colour disappears.

Bismuth tetroxide is best prepared by heating equal parts of bismuth oxide and potassium chlorate until the mixture ceases to glow, and then adding 2 parts of soda. The cold melt is washed with water until free from alkali, and the residue, which is a compound of sodium and bismuthic acid, agitated with 5 per cent. nitric acid. The dark, reddish-brown substance, which settles to the bottom is washed with water and dried. The composition of this compound, dried over sulphuric acid, is Bi_4O_8,H_2O. F. S. K.

Estimation of Free Oxygen in Water. By LATIEU (*Arch. Pharm.* [3], **26**, 128, from *J. Pharm. Anvers*, 1887, 570).—About 200 c.c. of water is treated with a definite quantity of sodium hydroxide and ferrous-ammonium sulphate solution in a narrow necked flask, closed by a perforated stopper. The precipitate formed is again dissolved by the addition of a definite volume of dilute sulphuric acid, and standard permanganate solution is added, corresponding with the excess of ferrous solution remaining. The excess of ferrous solution added above that indicated by the permanganate solution measures the free oxygen present. J. T.

Estimation of the Hardness of Waters. By E. v. COCHENHAUSEN (*J. pr. Chem.* [2], **37**, 413—416).—The author objects to

titrating a soap solution with calcium chloride or barium chloride, inasmuch as these salts are seldom or never found in natural waters, and have been shown to differ in their actions on soap solution from those that are generally contained in waters. The soap solution should be standardised by a solution of calcium sulphate, made either by diluting a solution saturated at a known temperature, and therefore of a known strength, or by titrating a saturated solution of lime water to neutrality with sulphuric acid of known strength, and diluting the calcium sulphate solution thus obtained until it contains an amount of sulphate equivalent to the quantity of calcium chloride generally employed. Before determining the hardness, the carbonates in the water are converted into sulphates by adding a slight excess of centinormal sulphuric acid, and titrating back accurately with centinormal alkali. A. G. B.

Detection of Impurities in Commercial Alcohols. By L. GODEFROY (*Compt. rend.*, **106**, 1018—1020).—6 or 7 c.c. of the alcohol is agitated with one drop of perfectly pure benzene, mixed with 6 or 7 c.c. of pure sulphuric acid of 66°, and again agitated. If reducing "head" products are present, the liquid immediately acquires a coloration which gradually darkens for a few minutes, and varies from pale brownish-yellow to black. Pure ethyl alcohol gives no immediate coloration, but after 8 or 10 minutes the liquid acquires a slight rose tint. This test will detect 1 c.c. of "head" products in 1000 litres of alcohol, or 1 part per 1,000,000. The quantity may be estimated by comparing the colour with that given by alcohol containing a known quantity of acetaldehyde, and expressing the results in terms of the latter.

If the liquid remains colourless after several minutes, no "head" products are present. In order to detect "tail" products, care is taken that the acid and alcohol are thoroughly mixed, in order to avoid polymerisation, and the liquid is boiled for a short time and then allowed to remain for several minutes. Under these conditions, pure ethyl alcohol gives an ochre-yellow coloration; but in presence of "tail" products the liquid acquires a brown colour with a green fluorescence, the depth of tint increasing with the quantity of impurity. This test is not so sensitive as the first, but will detect 1 part in 100,000.

Neither of these tests is directly applicable to wines, spirits, &c. These liquids should be distilled, and the first fraction tested for "head" products, and the last fraction for "tail" products.

C. H. B.

Estimation of Glucose by Fermentation. By GRÉHANT and QUINQUAUD (*Compt. rend.*, **106**, 1249—1250).—Estimations of glucose by fermentation are vitiated by the fact that carbonic anhydride is given off by the respiration of the yeast. If comparative experiments are made with equal quantities of yeast, the glucose being omitted in one case, the correction required can readily be determined, and the method then becomes accurate, and is especially suitable for dilute solutions which contain organic matter. Fermentation proceeds

equally well under atmospheric pressure or in a vacuum, but in the latter case the carbonic anhydride produced by the respiration of the yeast is, of course, much smaller. C. H. B.

Estimation of Reducing Sugar. By R. Gedult (*Chem. Centr.*, 1888, 201, from *Mon. Sci.* [4], **2**, 62—63).—This process, which dispenses with the filtration and reduction of cuprous oxide, depends on the decomposition of cuprous oxide by ammoniacal silver chloride according to the formula $2AgCl + Cu_2O = 2Ag + CuO + CuCl_2$, and the subsequent titration of the chlorine as $CuCl_2$ with silver nitrate after acidifying the solution. The preparation of standard solutions and the general mode of procedure is given in detail in the original. J. P. L.

Testing Beet-sugar for Purity. By A. Ihl (*Bied. Centr.*, 1888, 287).—Pure sugar dissolved in sodium hydroxide solution does not decolorise methylene-blue when boiled, whereas the smallest amount of invert sugar, grape-sugar, or dextrin, &c., decolorises methylene-blue. E. W. P.

Adulteration of Olive Oil. By R. Brullé (*Compt. rend.*, **106**, 1017—1018).—10 c.c. of the oil is mixed with 0·1 gram of dried powdered egg albumin and 2 c.c. of ordinary nitric acid, and the mixture is uniformly and gently heated. When the acid boils, the tube is inclined so that the albumin mixes with the oil, and if the latter is pure it acquires a greenish-yellow colour; but if mixed with not less than 5 per cent. of seed oils, it has an amber-yellow colour, and if the quantity of foreign oils approaches 50 per cent., the colour is deep orange. Nitric acid alone gives similar colorations, but only in presence of oils which are themselves coloured. The author has used this test with satisfactory results with earth-nut, sesame, poppy, colza, camelina, and linseed oils. Commercial so-called exotic aveline oil gives a rose tint with white streaks. C. H. B.

Estimation of Nicotine in Tobacco Extract. By J. Biel (*Arch. Pharm.* [3], **26**, 322, from *Pharm. Zeit. Russ.*, **27**, 3).—The extract, diluted with water, is treated with sodium hydroxide and distilled in a current of steam until the distillate no longer has an alkaline reaction. The distillate is faintly acidified with sulphuric acid, concentrated to one-tenth its volume, made alkaline with sodium hydroxide, and agitated with ether six times. The ethereal extract is evaporated to remove the ether and a little ammonia which may be present, the residue taken up with decinormal sulphuric acid, and the excess of acid titrated back with decinormal soda solution, using rosolic acid solution as indicator. About 7 per cent. of nicotine corresponds with a good extract. J. T.

Estimation of Caffeïne in Guarana. By A. Kremel (*Arch. Pharm.* [3], **26**, 318, from *Pharm. Post*, **21**, 101).—10 grams of guarana powder with 100 c.c. of 25 per cent. alcohol are placed in a

flask and the total weight noted. After digesting for one to two hours on the water-bath and cooling, the weight lost is made up with similar alcohol, and 50 c.c. (= 5 grams guarana) is filtered off, mixed with calcium hydroxide, and evaporated to dryness. The finely powdered residue is extracted with chloroform, from which extract the caffeïne is obtained in beautiful crystals on evaporation: these are dried at 100° and weighed. Commercial samples gave 3·12 to 3·8 per cent. of pure caffeïne. The ash of guarana amounts to 1·3 to 2·0 per cent., and is rich in phosphates. J. T.

Detection and Estimation of Magenta in Orchil and Cudbear. By C. Rawson (*Chem. News*, **57**, 165).—The following method is recommended as an improvement on the methods hitherto suggested for the detection and estimation of magenta in orchil and cudbear. 1 or 2 grams of cudbear, or an equivalent of orchil liquor, is boiled with 50 c.c. of alcohol, then diluted with 100 c.c. of water, and mixed with 15 to 20 c.c. of a strong solution of basic lead acetate, and a similar quantity of strong ammonia. The cudbear or orchil colouring matter is completely precipitated. On acidifying the filtered solution with acetic acid, the presence of magenta becomes apparent, and may be verified by testing; in this way 1 part of magenta may be detected in 100,000 parts of cudbear. The magenta is estimated colorimetrically by comparsion with a standard solution of pure magenta. D. A. L.

Two Methods for Detecting Vinoline. By E. Pollacci (*Chem. Centr.*, 1888, 86, from *Ann. Chim. Farm.*, **87**, 308—311).—The first method consists in mixing 6 to 7 c.c. of wine with nickel oxide to a thin paste, and allowing it to remain two to three hours. After this time, pure wine would be decolorised. The mixture is filtered, the residue washed with water, and then treated with 3 to 4 c.c. of a mixture of equal volumes of water, methyl and ethyl alcohols, strongly acidified with sulphuric acid. If the filtrate is red, vinoline is present.

The second process depends on the decomposition of the natural colouring matter of wine by lead peroxide, and the conversion of vinoline into a very yellow oxidation-product, which acids turn red, ammonia blue. The yellow colour changes to a beautiful violet after simple exposure to air. To test wine, 5 to 6 c.c. is mixed with lead peroxide, well stirred for two minutes and filtered. If the filtrate after the addition of hydrochloric acid remains colourless, vinoline is absent. J. P. L.

Detection of Picrotoxin in Beer, &c. By R. Palm (*J. Pharm.* [5], **17**, 19—20, from *Pharm. Zeit. Russ.*).—A solution of picrotoxin in either water or alcohol, when agitated with freshly-prepared lead hydroxide until the liquid has lost its bitterness, gives a stable compound between the alkaloïd and the lead. The least trace can be thus isolated, and the compound moistened with concentrated sulphuric acid gives a beautiful, saffron-yellow coloration, as well as if the alkaloïd were isolated in the pure state. It is essential that the

hydroxide should be freshly-prepared and well washed to free it from ammonia or potash. Lead carbonate, bismuth hydroxide, and other metallic hydroxides do not precipitate the alkaloïd. The suspected liquid is evaporated to dryness, the residue is taken up in very little water, slightly acidified, filtered, and shaken with ether. The residue from the ether is dissolved in a little water, filtered through animal charcoal, and the liquid completely precipitated by lead acetate, avoiding excess of this reagent. The filtered solution is finally well shaken with freshly-prepared lead hydroxide, and the yellow colour is obtained with sulphuric acid if picrotoxin is present. The colour persists several hours, and disappears on addition of an alkali, to reappear on addition of strong acid. J. T.

Estimation of Urethane in Urine. By G. Jacquemin (*J. Pharm.* [5], **16**, 538—539, from *J. pharm. Alsace-Lorraine*).—500 c.c. of urine is well shaken with ether, which is then separated and well washed to remove all trace of urine. The residue left by the ether on evaporation is dissolved in 10 to 20 c.c. of water, treated with potash in excess, and a standard solution of mercuric chloride is added until a persistent, yellow precipitate is produced. Urethane gives a more or less copious, white precipitate, but if in small amount, a yellow precipitate is formed which redissolves, and the amount of standard solution added when the yellow precipitate becomes persistent is proportional to the urethane present. J. T.

Estimation of Globulin. By Pohl (*J. Pharm.* [5], **16**, 536, from *Arch. f. ex. Path.*).—The author gives the preference to ammonium sulphate which he adds directly to serous liquids, and to acid urines after preliminary neutralisation with ammonia and separation of the phosphates by filtration. A saturated solution of ammonium sulphate is added to a urine thus neutralised, and after remaining an hour, the precipitate is collected on a tared filter, washed with ammonium sulphate solution, dried at 100°, and weighed. After ignition, the weight of the ash is deducted. J. T.

The Furfuraldehyde Colour Reaction. L. v. Udránszky (*Zeit. physiol. Chem.*, **12**, 355—376; compare this vol., p. 863).—Mylius (Abstr., 1887, 1149) has shown that the red colour produced by heating the bile acids, sugar, and sulphuric acid together, is due to the formation of furfuraldehyde from the two last-named reagents. He also showed that although of the substances he examined, the bile acids gave the test in the most marked manner, yet that there exist numerous organic substances that also give a similar reaction.

The present research is chiefly an expansion of Mylius' work. A very large number of organic substances were examined in the following way. A minute particle of the substance under investigation, or a drop of the substance if liquid, was placed in a test-tube with 1 c.c. of water or alcohol, and then a drop of solution of furfuraldehyde; concentrated sulphuric acid was added carefully, and the result watched. The temperature of the mixture was not allowed

to rise over 50°. As solutions of furfuraldehyde of a strength greater than 2·2 per cent. themselves give a coloration with sulphuric acid (showing an absorption-band spectroscopically at D), a solution of the strength 0·5 per cent. was used in all cases, which is a strength considerably greater than Mylius found necessary in the case of the bile acids. Some of the substances examined gave a coloration with sulphuric acid alone; the tint given with furfuraldehyde also varied a great deal; the particulars in each case are stated in a lengthy table. The reaction was found not to be a class reaction. The substances which give a colour are as follows:—Acetal, acetaldehyde, ethyl acetoacetate, acetone, ethylene glycol, malic acid, alizarin, amyl nitrite, anilidoacetic acid, aniline, anisaldehyde, anthracene, anthraquinone, apomorphine, atropine, benzaldehyde, borneol, catechol, brucine, quinic acid, cholesterin, cinchonine, codeïne, coniferin, conine, cumarin, cyanuric acid, cymene, digitaline, dimethylaniline, dihydroxytartaric acid, diphenylamine, gallic acid, Japan camphor, cresol, lævulinic acid, mesitylene, mesityl oxide, metaldehyde, methyl alcohol, methylhydantoïn, methylaniline, morphine, naphthalene, α-naphthol, α-naphthascatole, œnanthaldehyde, orcinol, paraldehyde, paraffin, phenanthrene, phenanthraquinone, phenol, phenylhydrazine, phloroglucinol, phorone, propaldehyde, protocatechuic acid, pyrogallol, resorcinol, salicaldehyde, salicylic acid, scatole, stearic acid, strychnine, toluene, thymol, tyrosine, valeraldehyde, vanillin, vanillic acid, vaselin, veratrine, metaxylene, paraxylene, cinnamaldehyde.

The substances which gave no colour are as follows:—Acetamide, acetanilide, acetophenone, alloxan, alloxantin, aspartic acid, benzonitrile, benzoic acid, succinic acid, hydrocyanic acid, pyruvic acid, butyric acid, caffeine, caproic acid, quinine, chinoline, quinone, quinoxaline, chloral hydrate, chloroform, citric acid, crotonic acid, cyanamide, dextrin, metadinitrophenol, dinitrotoluidine, dulcitol, acetic anhydride, formamide, fumaric acid, fermentation lactic acid, glycerol, glycocine, glycollic acid, glyoxal hydrogen sulphite, uric acid, urea, hippuric acid, isatin, leucine, malic acid, maltose, mandelic acid, mannitol, methylamine, orthonaphthoxyindole, metanitraniline, orthonitrobenzaldehyde, orthonitrobenzoic acid, orthonitrophenol, orthonitrophenylpropionic acid, oxalic acid, ethyl oxalate, parabanic acid, metaphenylenediamine, phenylacetic acid, picrotoxin, picric acid, piperidine, pyridine, quinol, mucic acid, starch, tannin, tetroxyethylbenzidine, grape-sugar, tartaric acid, trimethylamine, urethane, xylidine, cinnamic acid.

The spectroscopic appearances of the colours obtained differ in many cases; in all probability the products are therefore different; particulars in the case of a few of the more important substances are given; in the case of bile there is a band between D and E, and another at F. Several of the substances examined, for instance α-naphthol, give the test with greater delicacy than is the case with cholic acid.

The fact that the test is given by coniferin gave rise to a number of experiments on the colour reactions produced by strips of the wood of the pine and other trees.

In conclusion, the delicacy of the reaction as applied to the

detection of bile is discussed; using a 1 per cent. solution of furfuraldehyde in the manner already detailed, it is found possible to obtain a colour with 0·000033 gram of cholic acid; a quantity of 0·00005 gram gives a colour sufficiently intense to show its spectroscopic bands; in order to obtain evidence of the presence of bile acids in urine, it is generally necessary first to isolate them from that secretion. Normal urine does not contain bile acids. W. D. H.

Detection and Estimation of Indican and its Homologues in Urine. By W. MICHAILOFF (*J. Russ. Chem. Soc.*, 1887, 326—328).—Of all the methods for the detection and approximate determination of indican in urine in the form of indigotin, that of Senator, as modified by Salkowski, answers the best, but if the urine is too dark, it must be decolorised by the careful addition of basic lead acetate in order to remove urobilin; another disadvantage of this method is the great care which must be taken in the addition of calcium hypochlorite solution or of bromine-water. The author recommends the following method for the complete elimination of urobilin and its chromogen:—The acidified urine is saturated with finely powdered ammonium sulphate and repeatedly extracted with ethyl acetate. The aqueous liquid is then mixed with an equal volume of fuming hydrochloric acid, chloroform is added, and the oxidation performed with a very weak aqueous solution of bromine. In this way an extract is obtained containing indigotin compounds only and free from urobilin. No superoxidation can take place as in the case of the method recommended by Senator and Salkowski, and by the simultaneous oxidation and extraction with chloroform, the indigotin compounds are almost completely extracted. B. B.

Detection of Blood in Urine. By C. H. WOLFF (*Arch. Pharm.* [3], **26**, 125—126, from *Pharm. Centr.*, **28**, 637).—30 to 60 c.c. of urine is warmed on a water-bath with one-tenth its volume of a 3 per cent. zinc acetate solution. After 10 to 15 minutes, when most of the clear liquid can be poured off, the precipitate is washed on a small filter and dissolved over a test-tube with a little ammonia, followed by water, so that the total amount of liquid does not exceed 4 to 5 c.c. On this is placed a little benzene to exclude air, and two drops are added of a solution of tartaric acid and ferrous sulphate (1 gram each to 10 grams water). The reduced hæmatin can then be detected by means of a pocket spectroscope. J. T.

General and Physical Chemistry.

Refraction of Light by Ice, and by Water cooled below Zero. By C. PULFRICH (*Ann. Phys. Chem.* [2], **34**, 327—340).—The fact that whilst water attains its maximum density at a temperature of 4° its index of refraction decreases continuously from 0° to 100° is of considerable theoretical importance, as it shows that neither of the expressions $(\mu - 1)/D$ nor $(\mu^2 - 1)/D$ can be more than approximately constant.

The observations of Rühlmann (*Ann. Phys. Chem.*, **133**, 184) and of Damien (*Ann. l'école norm. sup.* [2], **10**, 275) have shown that the index of refraction diminishes again when water is cooled below 0°. The author finds that the index of refraction attains its maximum value at a temperature of about −1·5°. The author confirms Rühlmann's result that the curve representing the relation between the index of refraction and the temperature is not symmetrical on opposite sides of the maximum, resembling in this respect the similar curve for the density.

The author also describes some experiments on the double refraction of ice, and has made some measurements, using the method of total reflection, of the two principal indices of refraction for different rays of the spectrum. He finds that a sheet of ice 3 or 4 mm. thick cut parallel to the surface of solidification behaves like a slice of quartz $\frac{1}{2}$ mm. thick cut perpendicular to the axis. G. W. T.

Influence of Concentration on Fluorescence. By B. WALTER (*Ann. Phys. Chem.* [2], **34**, 316—326).—It is well known that the intensity of the fluorescent light emitted by a fluorescent solution increases up to a certain point as the dilution is increased, and then diminishes again. The author points out that in order for fluorescence to take place a certain quantity of light, A, must be absorbed, and of this a certain proportion, Fl, is reproduced by the phenomenon of fluorescence. When the solution is diluted, then if A remained constant Fl would be diminished, but A is also diminished by dilution. The author calls the ratio Fl/A the fluorescing power of the solution, and he has made a series of observations on solutions of different degrees of concentration of fluoresceïn, eosin, and Magdala red, from which he finds that this fraction increases without limit with the dilution of the solution. He considers it probable that the same law holds for all fluorescent substances, as it gives a satisfactory explanation of the apparently anomalous phenomenon referred to, and also of the fact that many substances which are strongly fluorescent in solution show no traces of fluorescence in the solid state.

In the case of a solid or of a highly concentrated solution the ratio Fl/A would be indefinitely small, so that no fluorescent effects would be observed, and, on the other hand, when the dilution increases beyond

a certain amount, although the proportion of light reproduced by fluorescence, to the amount absorbed, will continue to increase, the total amount absorbed will be so small that the fluorescence will be imperceptible. This explanation receives strong support from the fact that fluorescence can be observed in dilute solutions when the total absorption is too small to be measured.

The author considers that the fluorescence exhibited by some solid substances may be explained in the greater number of cases as being due to the dilution of the fluorescent substance by water of crystallisation or other transparent substance. G. W. T.

Fluorescence of Cupriferous Calcium Oxide. By L. DE BOISBAUDRAN (*Compt. rend.*, **106**, 1386—1387).—Calcium oxide which contains a small proportion of cupric oxide and has been calcined in presence of air, shows a very brilliant pale-green fluorescence in a vacuum, but no definite spectrum is observed. If the oxide is heated in hydrogen, the green fluorescence is replaced by a less intense rose or reddish fluorescence. With 0·1 per cent. of cupric oxide, the fluorescence is much more brilliant than with 1 per cent. When the former proportion is present, and the oxide is heated in hydrogen, the calcium-manganese band is seen in the spectrum of the fluorescence. With only 0·002 per cent. of cupric oxide, the fluorescence is more brilliant than with 0·1 per cent., but is white. Both green and red fluorescences diminish if the oxide is heated. The presence of manganese partially prevents the fluorescence due to copper.

C. H. B.

Spectral Analysis of Magnesium and Carbon. By A. GRÜNWALD (*Phil. Mag.* [5], **25**, 343—350).—The author uses the method of spectral analysis devised by him for oxygen and hydrogen (Abstr., 1887, 1070). The wave-lengths of the magnesium lines are divided into four groups. The first group, ascribed to the element "b" or helium, when multiplied by the proper factors, gives 21 lines, 15 of which agree with those of the water-spectrum of Liveing and Dewar; the rest divided by 2 give lines of the compound hydrogen-spectrum, and have probably not been observed in the water-spectrum owing to their feebleness. The second group of lines is that due to the element "c" in the same state as it occurs in oxygen. When multiplied by $\frac{3}{8}$ and $\frac{5}{8}$ these lines give lines of the water-spectrum, or lines whose wave-lengths are half those of lines of the compound hydrogen-spectrum. Only three lines due to "c" in oxygen are identical with those of the second group, the extinction of the rest being due to the influence of the other components of magnesium. The third group of magnesium lines is due to the element "b" in the state in which it occurs in hydrogen under the influence of "a" (Coronium). Multiplied by the proper factors these lines give 90 lines, which agree very closely with those of the water-spectrum. The fourth group consists of 50 lines, due to "b," as it occurs in hydrogen in water-vapour, nearly all of which correspond with lines observed by Liveing and Dewar. Only a few of the lines calculated for oxygen are found in the oxygen-spectrum, this being due, the author considers, to their feebleness.

Magnesium then is a compound containing (1) helium without condensation or dilatation, which in magnesium emits only the rays of Group I, all its other rays, including D_3, being weakened by the other components. (2.) The element "c," in the state in which it occurs in oxygen and carbon. (3.) The element "b," as it occurs in free hydrogen. (4.) The element "b," in the condensed state in which it occurs in the hydrogen of water-vapour.

Carbon examined in the same way is resolved into:—The element "c" as it occurs in oxygen and magnesium, and "b" in four different states: (1) in which its condensation is three-fifths of that which it has in hydrogen; (2) in the same state as it exists in hydrogen; (3) in which its condensation is five-fourths of that which it has in hydrogen; (4) in which its condensation is twenty-five-sixteenths of that which it has in hydrogen. H. K. T.

Electrochemical Actinometer. By GOUY and H. RIGOLLET (*Compt. rend.*, **106**, 1470—1471).—It is known that if copper with a superficial layer of oxide or basic salt is placed in a solution of copper sulphate, its electromotive force varies under the influence of light, but the phenomena are only distinct when the intensity of the light is high. The authors find that if copper coated with a layer of the oxide is immersed in a solution of a metallic chloride, bromide, or iodide, it is very sensitive even to rays of low intensity, and may consequently be used as an actinometer.

A carefully cleaned plate of copper is heated over a bunsen burner until the iridescence is replaced by a uniform tint. The superficial layer then consists mainly of cuprous oxide; if the heating is continued, cupric oxide is formed, and the plate is less sensitive. It may be protected by a layer of melted paraffin whilst cooling.

When a plate thus prepared is placed in a solution of sodium chloride together with a clean plate of copper, the electromotive force of the element is a few hundredths of a volt, the oxidised plate being positive. When exposed to light, the electromotive force increases, the oxidised plate becoming more strongly positive. Instead of plates of different kinds, two oxidised plates may be used, only one being exposed to light. The effect is instantaneous, and disappears when the light is cut off. Diffused daylight produces an alteration of several hundredths of a volt, and direct sunlight an alteration of at least a tenth of a volt. Gas and lamp flames also have a marked effect, and it would seem that the couple is sensitive to all the visible rays. The variation in the electromotive force is greater when the circuit includes a resistance of several hundred ohms. With a Thomson galvanometer, it is possible to recognise the effect of a candle flame at a distance of several metres.

Solutions of bromides behave in much the same way as chlorides, but in solutions of iodides, the couple is less sensitive.

C. H. B.

Electromotive Force produced by the Action of Light on Selenium. By W. v. ULJANIN (*Ann. Phys. Chem.* [2], **34**, 241—273).—The fact that an E.M.F. can be produced by the action of light on selenium was discovered by Adams and Day (*Proc. Roy. Soc.*, **25**,

113) who found that when one end of a plate of selenium, into opposite ends of which platinum wires had been melted, was submitted to the action of light, a current was produced, in general from the dark to the light electrode, but not infrequently in the opposite direction.

The subject was next investigated by Kalischer (*Rep. Physik.*, **17**, 563), who used an intermittent beam of light, and succeeded in obtaining sounds in a telephone in circuit with the selenium. Fritts (*Amer. J. Sci.*, **126**, 465) used a sheet of gold leaf as one of the electrodes, and illuminated the selenium through the transparent leaf. He succeeded in a good many cases in obtaining a measurable difference of potential between the ends of the selenium plate, and he found that the current arose immediately on exposing one of the electrodes to the action of light, and ceased instantaneously when the illumination ceased, but as long as the illumination was maintained constant, the current remained constant. He came to the conclusion that the current was due to the direct transformation of luminous into electrical energy. He also concluded from his experiments that there was no relation between the sensitiveness of different specimens to the effect of light, in altering the resistance and producing an E.M.F. respectively. His most sensitive specimens (with regard to the production of E.M.F.) were those in which polarisation was most marked.

Siemens (*Phil. Mag.* [5], **19**, 315) made some experiments with some of Fritts's specimens which had been forwarded to him, and confirmed his result with regard to the constancy of the current, and he also expressed his agreement with the conclusion that the current was due to the transformation of luminous into electrical energy.

The present investigation was undertaken at the suggestion of Kundt, principally with a view of finding a trustworthy method of preparing the specimens of selenium.

The specimens of selenium employed were tested by Rose, and were all found to contain traces of sulphur, whilst some of them also contained traces of mercury, antimony, arsenic, lead, bismuth, and copper. The author found a gold leaf electrode unsatisfactory, as it was very liable to peel off, and he ultimately used platinsed glass plates for both electrodes, connection being made with the circuit by means of strips of brass soldered to the platinum surfaces. These thin layers of platinum possessed the great advantage of allowing all the rays of the spectrum to pass with equal freedom. The plates were kept apart and parallel by means of strips of mica, and the selenium was melted in between them and cooled under pressure. In order to reduce the selenium from the amorphous to the crystalline form (in which only an E.M.F. is produced by the action of light) the plates were heated up to 195° in a paraffin bath and very slowly cooled. It was found that the repetition of this process increased the sensitiveness.

When it was desired to exclude the heat rays, the beam of light was made to pass through a solution of alum. When the plates were prepared in this manner, the difference in their behaviour was comparatively small, and no appreciable effect was produced even when 5 per cent. of sulphur was added to the selenium. The experimental results obtained were as follows:—

(1.) The effect of exposing one of the electrodes to the action of light was to produce an E.M.F. acting from the dark to the illuminated electrode. The E.M.F. disappeared completely when the source of light was shut off. The highest E.M.F. obtained was 0·12 volt.

(2.) In the case of plates prepared by other methods, some always gave an E.M.F. in the same direction whichever electrode was illuminated, but in these specimens its value was always greater when the negative electrode was the one exposed to the light.

(3.) When the electrodes were joined up through a metallic conductor the current flowed for hours from the dark to the illuminated electrode without undergoing any variation in strength.

(4.) When both poles were illuminated simultaneously, the E.M.F. could be weakened to any desired extent or reduced to zero.

(5.) The action of the light was found to take place instantaneously, and the current disappeared immediately and completely when both electrodes were darkened.

(6.) Many of the plates prepared were found to be subject to polarisation, but this was found to have no effect on the sensitiveness of the plates to the effect of light in producing E.M.F.

(7.) If the E.M.F. due to polarisation had attained a sufficient value, then the exposure of either electrode to a source of light diminished the E.M.F. With smaller values of the E.M.F., the effect of exposure to light was of the normal character described under (1). The limiting value of the E.M.F. of polarisation, below which the effects of exposure to light are of the normal character, varied greatly for different specimens.

(8.) The resistance of the plates and also their sensitiveness to the production of E.M.F. on exposure to light, in general diminished gradually with lapse of time. The initial resistances in the dark of the specimens employed, varied from 40,000 ohms to 900,000 ohms, and that of the last-named specimen gradually increased up to 1·4 megohm. In the case of plates prepared by the author's method, the effect of exposure to light on the resistance was to diminish it to about one-ninth of its value in the dark, a much greater sensitiveness than was exhibited by any of the plates prepared by other methods.

(9.) The resistance and also the sensitiveness to the production of E.M.F. were found to be considerably increased by passing alternate currents from a small induction coil through the plates, and plates which had lost their sensitiveness through lapse of time were restored to their former condition by the passage of alternate currents for a sufficient length of time. It was also found that in many cases, plates which previously behaved abnormally, could be reduced to the normal condition by the same method.

(10.) When the illumination was feeble, and the heat rays were excluded as completely as possible, the current was found to be proportional to the intensity of the light falling on the electrode, but when the illumination was stronger, and the heat rays were not excluded, the strength of the current increased more slowly than the illumination. In the latter case it would appear that the E.MF. increases more slowly than the conductivity.

(11.) When light from different portions of the spectrum was employed, the orange-yellow rays were found to produce the greatest effect in the case of a diffraction spectrum, and the greenish-yellow rays in the case of a prismatic spectrum.

The theory adopted by the author for the explanation of his results is as follows:—

He supposes, in the first place, that the selenium exists in the plates in several allotropic forms, some only of which are sensitive to the action of light. He further supposes that the plates contain particles capable of electrolysis, consisting either of combinations of different allotropic forms of selenium or of selenides of the metals present as impurities. He then assumes that the effect of exposure to light is in the first place to transform the sensitive particles into others which are electropositive to them, and in the second place, to facilitate the reunion of the electrolytic particles which have been broken up by the passage of the current. He then discusses the different experimental results, and shows that they may be explained on these assumptions.

G. W. T.

Study of Contact Electricity. By W. Ostwald (*Zeit. physikal. Chem.*, **1**, 583—610).—These investigations, undertaken with the view of ascertaining the potential differences between different metals and solutions, were carried out by means of dropping electrodes of mercury. Great difficulty was experienced in eliminating the potential difference between the electrodes themselves and the solution, and this could never be entirely accomplished, although it may be reduced to a minimum by proper adjustment of the aperture and regulation of the flow of mercury by its own pressure. The potential differences measured and tabulated are those between zinc, cadmium, tin, lead, iron, copper, bismuth, antimony, silver, and mercury, and solutions of hydrochloric, hydrobromic, hydriodic, nitric, sulphuric, phosphoric, formic, acetic, propionic, oxalic, and benzoic acids. The solutions used were normal, decinormal, and centinormal.

The potential difference between acid and metal depends of course in the first instance on the nature of the metal. For all the solutions examined, zinc and cadmium gave negative, copper, antimony, bismuth, silver, and mercury positive values, whilst tin, lead, and iron gave both positive and negative valves of 0·1 to 0·2 volt. The mean potential was for zinc −0·7 volt, cadmium −0·3, tin, iron and lead ±0, copper +0·3 to 0·4, bismuth +0·4, antimony +0·3, silver +0·5, and mercury +0·8. The nature of the acid has a very marked influence, the potential differences being generally smaller for the haloid acids than for the others. The influence of dilution appears generally to be in favour of increasing the negative potential and decreasing the positive one. At the same time the values obtained for the different acids approximate more closely to one another with increasing dilution.

A comparison of the potential differences between the above metals and acids with the heat developed in the reactions between them, shows that the order of these two values is to a great extent similar. If, however, from the heat developed the potential differences be calculated on the assumption that chemical is wholly converted into

electrical energy, the result is altogether unsatisfactory, and one that goes so far to prove that the above assumption of the total conversion of chemical into electrical energy cannot hold good. H. C.

Electrolysis. By W. W. H. Gee and H. Holden (*Phil. Mag.* [5], **25**, 276—283).—When a current passing between palladium electrodes is reversed, no gas is evolved at the pole which is now the anode, but streaks are seen descending through the liquid. After some time, the streaks disappear and gas begins to be evolved. Streaks are obtained if, before reversal, the electrodes are removed and cleaned, but not if they are heated to redness. They are not obtained if platinum electrodes are used. With phosphoric acid solutions, similar results are obtained. With sodium hydroxide solutions, descending streaks are obtained from the cathode without reversal, so long as the gas (hydrogen) is absorbed; when it begins to be evolved, the streaks are carried upwards. The authors consider the streaks to be due to the concentration of the electrolyte by electrolysis at one of the electrodes, the movement of the denser fluid being undisturbed so long as the palladium absorbs the evolved gas. If the electrodes are placed horizontally, ascending streaks can be easily observed at that electrode at which the electrolyte is being weakened, the lighter liquid no longer clinging to the metal. The dense streams were tested for palladium salts and hydrogen dioxide with negative results. A titration showed a higher percentage of sulphuric acid than there existed in the original electrolyte.

H. K. T.

Electrolytic Conductivity of Halogen Compounds. By W. Hampe (*Chem. Zeit.*, **11**, 1549—1550; **12**, 23—24, 106, 122, 140, 171, 173; compare this vol., p. 211).—Fused bismuth trichloride (m. p. 217°) is a very good conductor. The primary products of the decomposition are bismuth and chlorine, but the former acts at once on the trichloride to form the dichloride, hence the fused mass around the negative pole changes colour as soon as the circuit is closed, and in a short time the whole contents of the tube become brownish-black. When this is the case a portion of the chlorine liberated at the positive electrode combines with the dichloride to re-form the trichloride. On cooling, conductivity ceases with the solidification of the mass, and when the contents of the tube are treated with concentrated hydrochloric acid, an insoluble residue remains, consisting of very finely divided bismuth, produced by the decomposition of the dichloride; in most cases there are also some very small globules of the metal which has escaped conversion into dichloride.

Bismuth dichloride, prepared by fusing together the exact proportions of the trichloride and metallic bismuth, forms an opaque, black mass, which in very thin slices is transparent and dark-brown. It melts at 176° (uncorr.), and gives off vapour of the trichloride at 330°. It is decomposed by concentrated hydrochloric acid into metallic bismuth and the trichloride. In the fused state, it conducts as well as the trichloride, yielding the same primary products: the decomposition is much complicated by secondary reactions, with feeble currents the

liberated chlorine combines with the dichloride to form trichloride, with which the bismuth separated as a fine powder at the negative pole, re-forms dichloride. With stronger currents, a small quantity of chlorine escapes, whilst a corresponding quantity of bismuth is deposited, and provided the temperature has been sufficiently high (280°) is found as small globules.

Bismuth tribromide is a sulphur-yellow substance fusing at 217° to a red-brown liquid, and boiling at about 500°; the dibromide, prepared in the same way as the corresponding chloride, is a blackish-grey, crystalline substance melting at 202° (uncorr.) to a black liquid. These bromides behave chemically and electrolytically like the chlorides. As platinum is readily attacked, carbon electrodes were used in these experiments.

Molybdenum oxychloride (MoO_2Cl_2) is infusible at ordinary pressures. It forms a yellow solution with absolute ether, which does not conduct, but after agitating with a few drops of water, the current passes, and the negative pole becomes coated with molybdic molybdate. The sub-ethereal layer of water conducts readily on account of the hydrochloric acid present; under the influence of the current it becomes intensely blue, and very soon thickens to a magma. The oxychloride is very soluble in absolute alcohol, forming an acid solution, which conducts with evolution of hydrogen and formation of blue molybdic molybdate. The aqueous solution behaves like a hydrochloric acid solution of molybdic acid. Chlorine is evolved at the positive pole, whilst the reducing action of the liberated hydrogen is observed at the negative pole, so that the solution becomes first green, then blue, and some molybdenum molybdate separates, which, however, dissolves if much water is present. By the continued action of the current, molybdenum trichloride is formed, the solution becomes brownish-black and opaque in thick layers, and gives a black precipitate with potash. Green molybdenum oxychloride ($MoOCl_4$) does not conduct either in the fused state or in solution in ether, in which it dissolves to a red solution, becoming pale-yellow. Exposed to air it becomes covered with a red film, attracts moisture, and gives off hydrogen chloride. When quite pure it dissolves in water with a yellow colour, and the solution behaves towards the electric current like a hydrochloric acid solution of molybdic acid, but if the oxychloride ($MoOCl_4$) contains even the smallest quantity of tetrachloride, molybdenum molybdate forms, and the aqueous solution becomes blue in consequence. The violet oxychloride ($Mo_2O_3Cl_6$) dissolved in ether does not conduct, but in aqueous solution decomposes and behaves, with the current, like the other molybdenum oxychlorides.

Tungsten hexachloride, prepared by burning the metal in dry chlorine free from air, and purified by repeated sublimation, is a violet crystalline mass, which decrepitates on heating, melts at 275°, and boils at 346·7°. Tungsten pentachloride is readily crystalline, black in colour, but with green streak; m. p. 248°; b. p. 275·6°. Its vapour is greenish-yellow. Water decomposes it, with the formation of the bluish-black oxide. Tungstic tetra- and tri-chlorides are not fusible. The oxychloride, $WOCl_4$, forms red, transparent needles, melts at 210°,

and boils at 227·5°. The oxychloride, WO_2Cl_2, decomposes on heating. None of these tungsten compounds conduct.

Uranium tetrachloride is only obtained free from oxychloride by igniting an intimate mixture of uranous oxide and charcoal in a stream of dry chlorine, and allowing the products to pass over a long length of ignited charcoal before condensing in the drawn-out parts of the tube arranged for the purpose, where the tetrachloride collects, whilst the accompanying more volatile pentachloride passes on. The fused salt conducts excellently, chlorine is evolved, whilst metallic uranium is deposited at the negative pole, in peculiar irregular particles. After the action, the salt, when treated with water, evolves hydrogen, and yields a deep, dark-red solution, leaving a small quantity of uranium as an iron-grey powder. The red solution gives a black precipitate with ammonia, which, however, soon turns green with the evolution of hydrogen. The comparatively small yield of uranium is due to a secondary reaction, resulting in the production of the salt giving the red solution. Uranium oxychloride, UO_2Cl_2, conducts well, giving off chlorine with the separation of black, crystalline uranous oxide.

The only haloid oxygen compound adapted to these experiments is iodic anhydride; this substance does not conduct in ethereal solutions, but does in absolute alcohol. In this case, however, no gas is evolved, nor any iodine separated; it is therefore assumed that iodic acid and ether, and subsequently ethyl iodate are formed. Aqueous iodic acid is an electrolyte.

The chlorides of sulphur, S_2Cl_2 and SCl_2, do not conduct. Selenous chloride, Se_2Cl_2, when perfectly dry, scarcely conducts at all. Selenium tetrachloride sublimes without melting, and in ethereal solution does not conduct; absolute alcohol decomposes it, forming two layers, a heavy, oily, clear liquid, with a pale-yellow supernatant liquid, which reacts acid, and conducts the current moderately well, hydrogen being evolved at the negative pole, but as no selenium separates it is supposed to form some organo-selenium compound.

Tellurium dichloride melts at 160° (uncorr.) to a brownish-black liquid, which is an excellent conductor, with distinct polarisation current. The primary products of electrolysis are obscured by secondary reactions; the tellurium liberated at the negative pole dissolves in the dichloride, whilst the chlorine at the positive pole forms tetrachloride with the dichloride, and also dissolves; ultimately the tellurium and the tetrachloride re-form the dichloride. Tellurium tetrachloride melts at 209°, and is a good conductor; chlorine is evolved, and tellurium is precipitated as a black, flocculent powder; some dichloride forms as a secondary product. Tellurium di-iodide melts at 160° to a black liquid, and is a good conductor; iodine and tellurium are set free. On solidifying, the dichloride forms a lead-grey mass, with metallic lustre where it comes in contact with the glass walls of the vessel.

Manganous chloride melts at a red heat to a reddish-yellow liquid; is a good conductor; chlorine and manganese separate, the latter with indication of crystallisation.

The author, referring to the non-conductivity of water and hydrochloric acid individually, and to their conductivity when mixed,

inclines towards Thomsen's surmise, that the water and hydrochloric acid combine to form the compound H·ClH·OH, which on electrolysis breaks up into $\overset{+}{H}\ \overset{-}{ClH}\cdot OH$. The negative complex group may form either chlorine and water, or regenerate hydrochloric acid with the liberation of oxygen. Experimental evidence supports the latter view.

Dry fused ferrous chloride is an excellent conductor. Ferric chloride dissociates on heating, producing some ferrous chloride, and therefore conducts, but absolutely dry ferric chloride in dry ether does not conduct; if, however, the chloride is in the slightest degree moist, or if a few drops of water be mixed with the ethereal solution, it becomes a conductor, and hydrogen is evolved at the negative pole, but no gas comes off from the positive pole; with more water, iron is deposited, but although hydrogen is evolved no ferrous chloride forms, as otherwise, owing to its insolubility in ether, it would be deposited on the negative pole, which is not the case. No chlorine is evolved, as it enters into secondary combination. Ferric chloride is therefore not an electrolyte, but when mixed with water it becomes one. This conversion the author suggests is due to the formation of a compound, $Fe_2\cdot Cl_6H_6\cdot O_6H_6$, corresponding to the compound supposed to be formed with hydrochloric acid and water. He supports this suggestion by many arguments, and by demonstrating its accordance with the experimental data. Electrolysis would break it up into the groups $\overset{+}{Fe_2}\ 6\overset{-}{(ClH\cdot OH)}$, which would fully explain the occurrence of many secondary reactions. Both cobaltous and nickelous chlorides, when fused, conduct the current very well with evolution of chlorine and deposition of the metal in crystalline form.

Ruthenium hexachloride, Ru_2Cl_6, is infusible and insoluble in ether, somewhat soluble in absolute alcohol with greenish colour; this solution conducts to some extent, but hydrogen only is given off at the negative pole. The aqueous solution of ruthenium hexachloride reacts acid and conducts well, evolving chlorine and hydrogen, and depositing the hydroxide $Ru_2H_6O_6$. Ruthenium tetrachloride is not known in the free state; the dichloride is infusible and insoluble, and could therefore not be used in these experiments, a remark which applies to rhodium chloride, prepared by heating that metal in chlorine, and to the insoluble iridium chloride, Ir_2Cl_6. Rhodium chloride, Rh_2Cl_6, was prepared by precipitating sodium rhodium chloride with soda, dissolving the precipitate in hydrochloric acid, &c. This retained some sodium chloride, and when fused at an incipient red heat proved such a good electrolyte and decomposed with such vigour as to take fire. Chlorine is evolved from the carbon positive electrode, whilst metallic rhodium separates at the negative electrode of platinum wire.

Palladic chloride decomposes too readily to be isolated. Palladous chloride is dissolved only in traces by ether, but is soluble in alcohol and in water. Both the latter solutions conduct well, with the separation of metallic palladium. At a red heat palladous chloride fuses, giving off some chlorine, and forming a corresponding amount of subchloride. The fused mass conducts well; the deposited palladium easily forms a thread, which soon extends from pole to pole, and as soon as the metallic circuit is complete the palladium thread glows.

Osmium hexachloride is not known in a free state. Osmium tetrachloride fuses readily, but does not conduct. Osmium dichloride sublimes without melting, but dissolves slightly in ether, more readily in alcohol, and very easily in water, the latter solution soon undergoes decomposition. The ethereal solution does not conduct; the alcoholic conducts slightly; the aqueous readily, and at the negative pole an osmous hydroxide is deposited, which dissolves in hydrochloric acid to a red solution. Platinic chloride, as is well known, decomposes on heating, and is only very slightly soluble in dry ether, nevertheless this solution conducts feebly. The solution of dry platinum chloride in absolute alcohol conducts well, with separation of platinum sponge. The aqueous solution is also an excellent conductor, crystalline platinum separating.

Germanium chloride, $GeCl_4$, is a clear, colourless, mobile liquid of sp. gr. 1·887, and boiling at 86°; it does not conduct either at the ordinary temperature or at the boiling point. D. A. L.

Determination of the Size of the Molecules of Salts from the Electrical Conductivity of their Aqueous Solutions. By P. Walden (*Zeit. physikal. Chem.*, **1**, 529—549).—Ostwald (this vol., p. 331) has shown that if m is the molecular conductivity of a dilute salt solution, and v the dilution, dm/dv is of the form n_1n_2 const., where n_1 is the valency of the acid, and n_2 that of the base. The present paper contains further experimental proof of this view, and the results of the examination of a large number of salts.

The first portion of the paper contains the results obtained with magnesium salts. The salts of monobasic acids are found to behave very similarly to salts of monad bases with bibasic acids, the increase in the molecular conductivity with increase in the dilution from 32 to 1024 being between 17 and 20. Salts with bibasic acids give an increase of about double this amount, as was to be expected. The salts of fumaric and maleic acids differ somewhat, the former giving 32·7, and the latter 38·7, and a similar difference exists between the salts of mesaconic and itaconic acids, of which the first gives 30·2, the second 36·5. From this a similar structure may be argued for fumaric and mesaconic acids on the one hand, and for maleic and itaconic acids on the other. The salts of the tribasic citric, and aconitic acids form total exceptions from Ostwald's rule, and the tetrabasic ferrocyanide gave a number which is somewhat low.

In addition to the salts of magnesium, a number of other salts were examined. Potassium ferricyanide gave numbers which show the acid to be tribasic, so that the formula should not be doubled. Barium dithionate gives evidence of the acid being bibasic, and in the hypophosphite the acid is a monobasic one. Of the three sodium orthophosphates, only the monosodium salt gives a normal value, and the author concludes that it alone is undecomposed in solution. The pyrophosphate also appears to be decomposed in solution, and from the measurements obtained so too are potassium dichromate and sodium pyrosulphite, in the last of which, however, the bibasic character of the acid may be recognised. H. C.

Crystallisation of Salts during the Electrolysis of their Solutions. By S. PAGLIANI (*Chem. Centr.*, 1888, 437, from *Atti. R. Inst. Veneto* [6], **5**, 1181—1205).—During the electrolysis of solutions of copper, zinc or ferrous sulphates the salt crystallises on the anode, when this is made from the corresponding metal and does not exceed 40 sq. cm. per ampère. When concentrated solutions are used, the strength of the current at the anode is not the same for the different salts. The maximum value of the strength of current, with which this phenomenon occurs, is, for differently concentrated solutions of one and the same salt, the greater the more dilute the solution is. This value depends also very much on the temperature, as it increases as the latter rises. The strength of the current at the cathode is of no influence. J. W. L.

Magnetic Properties of Nickel. By H. TOMLINSON (*Phil. Mag.*, [5], **25**, 372—379).—A nickel wire is placed in the centre of primary and secondary coils consisting of copper wires surrounded with pipeclay and placed in an air-bath, whose temperature is determined by the resistance of a platinum coil. Similar primary and secondary coils without nickel wire are maintained at a constant temperature and connected in series respectively with the above coils and with a battery. The coils are so arranged that the induced currents neutralise one another when the nickel wire is omitted. In making an experiment, when a fixed temperature is reached, the resistances of the coil are made equal to what they were when cold, and the current in the primary is adjusted and opened and closed until the deflections of a reflecting galvanometer become equal. The magnetising force was finally raised to 18·183 C.G.S. The induced currents were compared with those of an earth-coil rotating on a horizontal axis. It was found that the temperature of maximum permeability is lower the greater the magnetising force, whilst the temperature at which the permeability vanishes is higher the greater the magnetising force. The nickel wire used contained iron. A brass wire coated with pure nickel when heated and placed between the poles of an electromagnet, set itself axially with great suddenness as the wire cooled. The rate of loss of permeability appears to be greatest at a temperature of 300°. H. K. T.

Recalescence of Steel. By H. F. NEWALL (*Phil. Mag.* [5], **25**, 510—512).—A steel wire heated by an electric current in a vacuum and in nitrogen gas showed unaltered recalescence, proving that the phenomenon is not due to chemical action or to occluded gases. Recalescence is also not due, as suggested by Forbes, to unequal conductivity at different temperatures, since a steel wire (0·5 mm. in diameter) hammered out to 0·1 mm. thickness still showed reglow. This conclusion is also borne out by an experiment in which a steel rod was allowed to cool slowly, the temperature of the interior being ascertained by means of a platinum-copper couple. At the moment of recalescence, the galvanometer showed a rise of temperature. Similarly when the rod was slowly heated, a fall of temperature was observed at the moment of darkening. The temperature of darkening is higher than that of recalescence, hence the electromotive

force produced when a flame is moved along a steel wire heated so as to show reglow behind the flame and darkening in front of it.

H. K. T.

Molecular Heat of Solid Compounds. By H. Kopp (*Ber.*, **21**, 1880—1882).—The author has already (Abstr., 1879, 769) pointed out that the elements carbon, boron, and silicon form exceptions to Dulong and Petit's law, whilst many compounds do not conform to the laws of Neumann and of Garnier and Cannizzaro, when the specific heats are considered as found for the compounds in the solid state and nearly at the ordinary temperature. It was suggested that as the elements carbon, boron, and silicon obey Dulong and Petit's law at a higher temperature, the same might hold good for compounds. This view is supported by recent experiments made with quartz, which is shown by Pionchon (*Compt. rend.*, **106**, 1344) to have the sp. heat = 0·305 at 400° to 1000°; sp. heat at 0° = 0·1737. The mol. heat of silica (60 × 0·305 = 18·3) is nearly the same as those of analogously composed metallic chlorides:—$BaCl_2$ = 18·6 − 18·8, $CaCl_2$ = 18·2, &c. The number 18·3 when divided by 3 gives the average atomic heat = 6·1. N. H. M.

Heats of Combustion of Fumaric, Maleic, and the Pyrocitric Acids. By W. Louguinine (*Compt. rend.*, **106**, 1289—1291).—The results obtained by means of the calorimetric bomb were as follows:—

Fumaric acid	318·176	cal.
Maleic acid	331·702	„
Mesaconic acid	479·063	„
Citraconic acid	477·867	„
Itaconic acid	476·58	„

The difference between the heats of combustion of fumaric and maleic acids is considerable, and it is evident that these acids must differ profoundly in their constitution.

In the case of the pyrocitric acids the differences are small, and it follows that the differences in their constitution must be much less profound than in the case of the first two acids.

Fumaric acid is the true homologue of the pyrocitric acids.

C. H. B.

Heat of Combustion of the Solid Isomeride of Benzene. By W. Louguinine (*Compt. rend.*, **106**, 1472—1473).—The solid isomeride of benzene obtained by Griner (Abstr., 1887, 1033) was burnt in the form of small pastilles in the calorimetric bomb. Heat of combustion for 1 gram = 10863·9 cal. or for 1 gram-molecule 847384 cal. This value approximates somewhat closely to that obtained for gaseous propargyl (853600 cal.), a result which confirms Griner's supposition that they are of similar constitution, thus: Dipropargyl, $CH{:}C{\cdot}CH_2{\cdot}CH_2{\cdot}C{:}CH$, and the solid isomeride, $CMe{:}C{\cdot}C{:}CMe$. The heat of combustion of benzene is much lower and its constitution is essentially different. C. H. B.

Heat of Neutralisation of Ethyl Cyanomalonate, Acetocyanacetate, and Benzoylcyanacetate. By A. HALLER and A. GUNTZ (*Compt. rend.*, **106**, 1473—1476).—The heats of neutralisation were determined directly, or by decomposing the sodium or barium derivative with sulphuric acid, which precipitates the ethereal salt.

Ethyl Cyanomalonate.—Heat of neutralisation by barium oxide $+2 \times 15{\cdot}0$ Cal.; by sodium hydroxide $+15{\cdot}2$ Cal. These values are higher than the heat of neutralisation of hydrochloric acid and almost as great as that of sulphuric acid. It follows that ethyl cyanomalonate is a strong acid. Its sodium salt is not completely decomposed by sulphuric acid, but equilibrium is established between the acid, the ethereal salt and their respective sodium-derivatives. The barium salt crystallises with 4 mols. H_2O. The heat of dissolution of the anhydrous salt is $+2{\cdot}4$ Cal.; that of the hydrated salt $-4{\cdot}9$ Cal., hence the heat of formation of the solid hydrate from the salt and solid water is $+1{\cdot}6$ Cal. The heat of dissolution of the sodium salt at 10° is $-2{\cdot}6$ Cal.

Ethyl Benzoylcyanacetate.—Heat of dissolution of the sodium-derivative $-2{\cdot}75$ Cal. If the solution is mixed with sulphuric acid, the ethereal salt is precipitated, and if sodium hydroxide is added immediately, the precipitate dissolves with development of $+11{\cdot}2$ Cal. If instead of adding the alkali the liquid is allowed to remain about 10 minutes, the precipitated ethereal salt changes in appearance and properties, with development of $+3{\cdot}6$ Cal. The heat of neutralisation as determined by treatment with sulphuric acid is $+11{\cdot}6$ Cal. If the heat of transformation of the ethereal salt is taken into account, it is found that the heat of neutralisation of the crystallised ethereal salt is $+7{\cdot}85$ Cal. From these results it follows that the acid function of ethyl benzoylcyanacetate is less energetic than that of ethyl cyanomalonate.

Ethyl Acetocyanacetate. — Heat of neutralisation by sodium hydroxide $+10{\cdot}5$ Cal. The precipitated ethereal salt undergoes a transformation similar to that of ethyl benzoylcyanacetate with development of $+1{\cdot}75$ Cal. The acid function of this ethereal salt is more energetic than that of the preceding salt.

It is evident that the acidic function of the ethyl salts of substituted cyanacetic acids is comparable with that of formic, oxalic, picric, and tartaric acids. C. H. B.

Specific Gravity of Aqueous Solutions. By T. GERLACH (*Zeit. anal. Chem.*, **27**, 271—358).—In groups of salts containing the same acid and of similar constitution, the specific gravity of solutions of equal molecular concentration (that is, containing an equal number of molecules per unit weight of water) increases with the molecular weight of the dissolved salt. The contraction which occurs during the solution of a solid is generally least for the salts of greatest solubility, but to this there are many exceptions, and in the case of certain ammonium salts, expansion even occurs during solution. The contraction is invariably greater with strong than with weak solutions. The molecular heat of salt solutions is (with the exception of a strong solution of sodium acetate) less that the sum of the molecular heats

of the water and salt separately. This modification of the molecular heat is, as a rule, less with strong solutions than with weak ones. For solutions of equal molecular concentration the amount of the modification increases with the molecular weight of the dissolved salt. The value of the fraction $\frac{1 - \text{sp. heat}}{\text{sp. gr.} - 1}$ increases with increased dilution. The quotient obtained by dividing the specific gravity by the specific heat increases with increase of concentration. For equal molecular concentration this quotient increases with the molecular weight of the salt. M. J. S.

Molecular Volumes of Aromatic Compounds. By F. NEUBECK (*Zeit. physikal. Chem.*, **1**, 649—666).—The molecular volumes of a series of related compounds were determined under different pressures at the corresponding boiling points. The substances thus examined were benzene, toluene, xylene, and their nitro- and amido-derivatives. A series of experiments was made with each compound at pressures varying from 150 to 760 mm., and in each case a curve was drawn with pressures as abscissæ and molecular volumes as ordinates. These curves show that an increase in the pressure is attended by an increase in the molecular volume, so that if the pressure increase from P to ΔP, there will be a corresponding increase of Δv in the molecular volume, but always so that in the units taken Δv is smaller than ΔP; or, in other words, the molecular volume increases less rapidly than the vapour-tension.

A divergence of the curves as the distance from the axis of ordinates is increased, shows that the difference between the molecular volumes of two compounds becomes greater with rising pressures. But by a comparison of these differences for different pressures, it is seen that those regularities which have been established for one pressure still hold good at any other.

It further appears that the influence which an additional substituting group has on the molecular volume of a benzene compound, depends on the position which it takes up relatively to the group already present. If it takes up a para-position, its influence is about the same as when entering into benzene alone, but it is greater than this in the meta-position, and greater still in the ortho. The assumption that each constituent of a compound has a definite molecular volume is not absolutely correct, and therefore, also, the representation of the molecular volume of a compound as the sum of those of its constituents. Among isomeric compounds, the para have the greatest and the ortho the least molecular volumes, and the difference between those of the para and meta is less than between those of the meta and ortho. H. C.

Molecular Constitution of Dilute Solutions. By M. PLANCK (*Zeit. physikal. Chem.*, **1**, 577—582; compare this vol., p. 780).—In a former paper (*Ann. Phys. Chem.* [2], **32**, 499) the author has shown that if in any dilute solution n is the number of molecules of the dissolved substances, n_0 the number of molecules of the solvent, θ_0 the freezing point of the solvent, and θ that of the solution in absolute

temperature, and finally Q_0 the latent heat of fusion of one molecule of the solvent, the lowering of the freezing point of the solution is $\theta_0 - \theta = \theta_0^2 n/Q_0 n_0$. From this expression the value for the molecular reduction of the freezing point may be deduced, $T = M(\theta_0 - \theta) = 1{\cdot}97\ \theta_0^2 nM/q_0 n_0 M_0$, where q_0 is the latent heat of fusion and M_0 the molecular weight of the solvent, and M the molecular weight of the dissolved substance. The molecular reductions thus calculated for acetic and formic acids, benzene, nitrobenzene, and water agree with those observed by Raoult.

The author further points out that the expression nM, which should be equivalent to the mass of the dissolved substance, will only be correct as long as that substance possesses the molecular weight M. If combination or dissociation of the molecules originally present take place, then the mass of the dissolved substance becomes generally nM/i, where i is less than unity if combination takes place and greater than unity in the case of dissociation. The introduction of i into the above expression leads to results similar to those obtained by Van't Hoff (this vol., p. 780). H. C.

Dissociation of Substances Dissolved in Water. By S. Arrhenius (*Zeit. physikal. Chem.*, **1**, 631—648).—Exceptions to Van't Hoff's law for osmotic pressure in solutions (this vol., p. 778), in cases where the pressure is greater than it theoretically should be, may be explained on the assumption that the molecules of the dissolved salt have undergone dissociation. In the case of electrolytes, the Clausius hypothesis assumes a partial dissociation of the salt into its ions to have taken place, and could it be ascertained to what extent, it would still be possible to apply Van't Hoff's law for calculating the osmotic pressure.

The author has termed the molecules of an electrolyte which may be assumed to exist dissociated in solution the "active" molecules, and the rest which remain intact the "inactive," and the ratio between the number of active molecules and the sum of the active and inactive, the "activity coefficient," α (Lodge's dissociation ratio). This number is the ratio between the actual molecular conductivity of the solution and the limiting value which it approaches with increasing dilution. α being known, Van't Hoff's coefficient i, the ratio between the osmotic pressure which a substance exercises and that which it would exercise if composed solely of inactive molecules, may be calculated. For i is equal to the sum of the inactive molecules and the number of ions, divided by the whole number of active and inactive molecules. Hence if m is the number of inactive and n that of active molecules, and k the number of ions into which each active molecule separates, then $i = (m + kn)/(m + n)$ or since $\alpha = n/(m + n)$ then $i = 1 + (k - 1)\alpha$. The other method of calculating i is that given by Van't Hoff, and consists in dividing the observed molecular reduction in the freezing point of water produced by the substance by 18·5.

A table is drawn up of the values of i for a large number of salts calculated by both the above methods, and it shows a general agreement between the two. From this the author argues the truth of his

assumption of the dissociation of substances dissolved in water, on which the calculation is based. He concludes that Van't Hoff's law holds for all substances, the exceptions being due to dissociation, and that dissociation of electrolytes always takes place in solution, increasing in amount with the dilution until in an infinitely dilute solution only active molecules occur.

In the second portion of the paper he enters into a discussion of the other properties of solution with reference to their bearings on the above views. H. C.

Hydrates of Gases. By H. W. B. Roozeboom (*Compt. rend.*, **106**, 1292—1293).—A claim for priority over de Forcrand and Villard (this vol., p. 644). The author's papers may be found in *Rec. Trav. Chim.*, vols. **3**, **4**, **5**, and **6**. C. H. B.

Hydrates of Hydrogen Sulphide and Methyl Chloride. By de Forcrand and Villard (*Compt. rend.*, **106**, 1402—1405).—The apparatus employed for the determination of the composition of these compounds consists of a strong bulb of 150 c.c. capacity, into the neck of which is fused a vertical tube reaching to the bottom of the bulb. Attached laterally to the neck of the bulb is a bent tube with a bulb of 15 to 16 c.c. capacity, terminating in a tube drawn out to a point. 40 to 50 grams of mercury and 0·2 to 0·6 gram of water is introduced into the side bulb and some mercury is poured into the larger bulb until it closes the opening of the vertical tube. A current of the gas is passed into the apparatus until all the air is expelled; the drawn-out end of the bulb is sealed, and the passage of the gas is continued until the apparatus is filled with gas at a pressure of a few centimetres above that of the atmosphere. It is then cooled to 0° and the pressure of the gas measured. A part of the side bulb is now cooled below 0°, and crystals of the hydrate begin to form. Mercury is poured down the vertical tube, from a quantity previously weighed, in order to maintain the pressure, and the mercury in the side bulb is repeatedly agitated in order to break up the layer of crystals. When the crystals have become quite dry, the absorption of gas ceases, and after the height of the mercury has remained constant for several hours, the pressure is readjusted. The volume of gas absorbed is measured by the quantity of mercury which has been added. The apparatus is now placed in a warm room, when the crystals melt with evolution of gas, which expels the mercury through the vertical tube. The side bulb containing mercury and water is detached and weighed, then dried and weighed again, the loss in weight giving of course the quantity of water.

In this way it was found that the hydrate of hydrogen sulphide has the composition $SH_2 + 7H_2O$, and the hydrate of methyl chloride the composition $MeCl + 9H_2O$. C. H. B.

Hydrate of Methyl Chloride. By de Forcrand and Villard (*Compt. rend.*, **106**, 1357—1359).—The following values were obtained for the tension of dissociation measured from 760 mm.:—

Temp....	0·0°	0·5	0·9	2·4	4·8	6·5	8·0	10·5
Pressure..	−449	−426	−411	−344	−203	−73	+6·5	+336

Temp.....	12·5°	14·35	14·5	14·9	15·5	16·2	17·5	19·5
Pressure..	+648	1065	1083	1208	1368	1513	1920	2382

Temp.....	20·1°	20·4	21·0	22·0
Pressure..	2750	2927	2996	3134

The tension is equal to 760 mm. at +7·3. Up to 20·6 only water and crystals of the hydrate are present, but at this temperature some of the methyl chloride separates in oily drops on the surface of the water, although some crystals of the hydrate remain undecomposed. At 20·9 the crystals are entirely decomposed and the drops of methyl chloride volatilise. The curve representing the tensions of the hydrate and Regnault's curve for the tension of methyl chloride intersect at about 20·5. C. H. B.

Osmose through Precipitated Diaphragms. By G. TAMMANN (*Ann. Phys. Chem.* [2], **34**, 299—315).—If a solution is separated from a portion of the same solvent by a precipitated diaphragm which does not allow the substance in solution to pass through it, then the osmotic coefficient of the solution is represented by the quantity of the solvent which flows through unit area of the diaphragm in unit time. If at the temperature of observation, the vapour-pressure of the dissolved substance is zero, the values of the osmotic coefficient of the solutions, with similar diaphragms, will be proportional to the amounts by which their vapour-pressures are lowered. From this it would follow that solutions having equal osmotic coefficients would also have equal vapour-pressures. The author has been for some time engaged on a series of experimental investigations with a view of testing the truth of this relation, and in the meantime Van t'Hoff has shown (this vol., p. 778), from thermodynamic considerations, that no osmose can take place between solutions which have equal vapour-pressures.

The author found that Pfeffer's method of observation (*Osmotische Untersuch.*, 1887, 15) was not trustworthy, owing to the liability to change in the state of the diaphragm. The method employed in the present research is based on the observation of phenomena depending on changes in refrangibility of portions of the solutions on opposite sides of the diaphragm, due to changes in density produced by osmotic currents.

A large number of diaphragms were tried, but the only satisfactory ones consisted of ferrocyanide of zinc and ferrocyanide of copper formed by the contact of salts of zinc or copper with solutions of ferrocyanide of potassium.

Some of the experiments seemed to indicate that the vapour-pressure of a solution of potassium ferrocyanide is increased when a portion of zinc ferrocyanide is dissolved in it.

Both the diaphragms were found to be permeable to potassium chloride and nitrate, but impermeable to the sulphate.

The copper ferrocyanide diaphragm was also found to be permeable, against the direction of the osmotic current, to carbamide, ethyl and propyl alcohols, and ethyl acetate; and impermeable, against the direction of the current, to barium chloride, ammonium sulphate, cane- and grape-sugar, salicin, isobutyl alcohol, and chloral hydrate.

From experiments on a number of mixtures, the author found that

in general the lowering of the vapour-pressure was sensibly equal to the sum of the amounts by which the vapour-pressures of the constituents would be lowered separately, but there were some decided exceptions to the rule, namely, the lowering of the vapour-pressure of mixtures of ammonium sulphate and copper sulphate, barium chloride and copper chloride, and potassium chloride and copper chloride was found to be greater than it would be if this relation held good. The author illustrates the results of his experiments by a number of isosmotic curves, in which the ordinates represent the molecular concentration of the solution of potassium ferrocyanide, and the abscissæ the molecular concentration of the salt compared with it. In the case of simple metallic salts, he finds that these curves agree with similarly drawn curves of equal vapour-pressure, proving the truth of the law that solutions which have equal osmotic coefficients have equal vapour-pressures.

The isosmotic curves for the different organic substances (dissolved in solutions of copper sulphate) investigated were found to agree very closely, and their deviations from a mean curve representing the whole of them were within the limits of experimental error.

This seems to show that the osmotic coefficient does not depend on whether the substance in solution is a solid or a liquid, but this is not true for the vapour-pressure, and therefore a solution of potassium ferrocyanide cannot have the same vapour-pressure as isosmotic solutions of ethyl ether, ethyl alcohol, and propyl alcohol. If then two solutions are isosmotic the molecular motions of the common solvent must be equal, and if the substance in solution is a solid the vapour-pressure may be taken as a measure of the molecular motion, so that isosmotic solutions of such substances have equal vapour-pressures. If a liquid is dissolved in a liquid, then at the freezing point the vapour-pressure of the solution and the solidified solvent will certainly not be equal, but the molecular motion of the solvent must be the same for the solid and liquid portions, and therefore isosmotic solutions of liquids must have equal freezing points.

If the lowering in the freezing point and in the vapour-pressure of a solution are proportional to the percentage of substance in solution, the quotient of the molecular lowering of the freezing point of two solutions must be equal to that of their isosmotic concentration, and the author gives a table comparing the latter quotients obtained from his own experiments with the quotients of the molecular lowering in the freezing points according to Raoult (*Compt. rend.*, **94**, 1517), the values for potassium ferrocyanide being taken as the standard of comparison, and the agreement appears to be sufficiently close to justify the conclusion. G. W. T.

Constitution of Solutions. By F. Rüdorff (*Ber.*, **21**, 1882—1885; compare this vol., p. 342).—Diffusion experiments made with 6 per cent., 10 per cent., and 13·5 per cent. (saturated) solutions of potassium copper sulphate show that the decomposition of the double salt does not depend on the degree of concentration, the decomposition being nearly the same in each case. If, however, coarsely powdered salt is put into the diaphragm so that the solution

will remain concentrated, or nearly so, a different result is obtained; the diffusate contained to 100 parts of copper an average of 183·9 of potassium instead of 287·0, 273·9, and 280·8, obtained as means of the three other experiments. With solutions of ammonium magnesium sulphate, potassium chrome alum, ammonium manganese sulphate, and ammonium cupric chloride, similar results were obtained. N. H. M.

Absorptive Power of Colloid Substances. By J. M. VAN BEMMELEN (*Landw. Versuchs-Stat.*, **35**, 69—136). See p. 985.

Rate of the Reaction between Marble and Hydrochloric Acid. By J. J. BOGUSKI (*Zeit. physikal. Chem.*, **1**, 558—564).—The author finds that experiments conducted at different temperatures cannot be compared on account of the change in the surface of the marble. At a temperature of about 19—21° the rate of the reaction is proportional to the concentration of the acid, at 0° it increases more slowly, at 50° more rapidly than the concentration. Thus if v be the rate of the reaction and y the concentration, at 20° $v = ky$, at 50° y must be raised to some power greater, at 0° to one less than unity.

Much appears to depend on the viscosity of the solution. Thus the reaction is far less rapid with hydrochloric acid dissolved in glycerol than in water, and less rapid in water than in alcohol. H. C.

Rate of the Reaction between Iceland Spar and Acids. By W. SPRING (*Bull. Soc. Chim.*, **49**, 3—11).—The rate of the reaction between Iceland spar and acids was determined by the same method as that described in the case of marble (Abstr., 1887, 882). The volume of acid employed was such that the reaction ceased after the evolution of 522 c.c. (760 mm., 15°) of carbonic anhydride. The spar was examined (1) in the direction of cleavage, (2) in a direction parallel with, and (3) in a direction perpendicular to the principal axis, with the following results:—

(1.) All surfaces of cleavage dissolve with the same rapidity when treated with 10 per cent. hydrochloric acid at 15°, 35° and 55°, other conditions being equal. Between 15° and 35° the rapidity of solution varies in proportion to the concentration of the acid, but at 55° the rapidity diminishes more quickly than the concentration. At each of the three temperatures, after the evolution of 350 c.c. of carbonic anhydride, the rapidity of solution diminishes so quickly that practically the reaction may be considered as null after the evolution of 400 c.c. at 15° and 35°, and 425 c.c. at 55°; the concentration of the acid is then 2·34 per cent. and 1·86 per cent. respectively. Iceland spar is only attacked with extreme slowness by 2 per cent. hydrochloric acid. For each of the three temperatures, the rapidity of solution is less than in the case of marble, and the difference increases slowly, but regularly, with the temperature. The law of solubility is the same for Iceland spar as for marble, the temperature being the same, but in the case of the former the reaction does not commence until the hydrochloric acid has attained a concentration of 2·34 per cent. Unless special precautions are taken, the rapidity of the reaction appears to

reach its maximum only after the production of 50 to 75 c.c. of gas, owing to absorption of gas by the liquid. The rapidity of solution is the same in hydrochloric, nitric, and hydriodic acids of equivalent strengths, but in hydrobromic acid the rapidity of solution exceeds that in the above-named acids in proportion to the concentration, so that the difference tends to become null as the concentration diminishes.

(2.) The rapidity of the reaction with faces cut parallel to the principal axis is, at 15°, practically the same as with faces of cleavage, but at 35° it is 1·23 times, and at 55° it is 1·28 times greater on the average, so that above a certain temperature the rapidity of solution no longer diminishes in proportion to the temperature.

(3.) With faces cut perpendicular to the principal axis, the rapidity of solution is greater than in the preceding case. At 15° the rapidity diminishes in proportion to the concentration, at 35°, and especially at 55°, the rapidity diminishes very slowly until about one-tenth of the theoretical quantity of gas has been evolved, the rate of solution then decreases more rapidly, and finally, after about one-fifth of the acid is used up, becomes proportional to the concentration of the acid. The rapidity of the reaction increases, therefore, during a certain time, in spite of a decrease in the strength of the acid, just as is the case with marble, and this phenomenon is not in any way due to the absorption of gas by the liquid at the commencement of the reaction.

From a comparison of the results obtained in the last two cases, it seems that a certain relation exists between the chemical activity of a substance and its optical elasticity in a given direction.

F. S. K.

Rate of Formation of Ethereal Salts. By N. Menschutkin (*Zeit. physikal. Chem.*, **1**, 611—630).—The determination of the rate of formation of acetates from different alcohols in benzene solutions, gave the following comparative values for the constants of velocity, that of methyl alcohol being taken as 100:—

Primary Alcohols.

Methyl alcohol	100·0	Octyl alcohol	35·8
Ethyl „	47·9	Tetradecyl alcohol	27·6
Propyl „	45·6	Hexdecyl „	25·5
Butyl „	44·1	Octodecyl „	23·2
Isobutyl „	38·1	Myricyl „	16·5
Heptyl „	37·3		

Unsaturated Primary Alcohols.

Allyl alcohol	27·2	Benzyl alcohol.....	26·6
α-Methylallyl alcohol..	25·2		

Secondary Alcohols.

Dimethyl carbinol	14·1	Methyl hexyl carbinol	8·7
Methyl ethyl carbinol .	11·6	Methyl allyl „ ..	6·1

Tertiary Alcohol.

Trimethyl carbinol, 0·8.

From this table it will be seen that the values for the constants are greatest for the primary alcohols, and that again among these they are greater for the saturated than for the unsaturated alcohols. In homologous series an increase in the molecular weight is found to be attended with a decrease in the value of the constant, which decrease is, however, not the same for each CH_2-group added.

The effect of varying the solvent in which the reaction takes place is to materially alter the value of the constant. Thus putting the value of the constant for isobutyl alcohol in benzene solution as unity, we get in xylene solution 1·37, and in hexane solution 2·18, and with the value for isopropyl alcohol in benzene as unity, we get in xylene 1·25, and in hexane 2·07. H. C.

Equivalents of the Elements. By DELAUNEY (*Compt. rend.*, **106**, 1405—1407).—If the equivalent of hydrogen is taken as unity, the equivalents of the other elements are represented by the expression $\frac{N}{3}\sqrt{5^2-n^2}$, in which N and n are whole numbers, the value of n being 0, 1, 2, 3, or 4. The calculated numbers agree fairly well with the actual determinations. The elements may be divided into groups or families according to the values of n, but the same element may fall into two or even three families. The omission of $\frac{N}{3}$ merely changes the unit of equivalence, and hence only the simplified expression $\sqrt{5^2-n^2}$ need be considered.

The author imagines the existence of a primitive molecule composed of 5 atoms revolving at different distances round a central atom after the manner of planets round a sun. If such a system is penetrated by a group of atoms, all tangent to one another, the opposite directions of their motions will give rise to a stress which will result in an agglomeration of the atoms. C. H. B.

A Certain Atomic Hypothesis. By K. PEARSON (*Cambridge Phil. Trans.*, **14**, 71—120).—This paper is one of very great interest to chemists, but in its original form would probably repel them by its masses of mathematical formulæ. The author points out that the view which regards the physical universe as a continuous medium, is rapidly displacing the old molecular hypothesis. Certain portions of this medium are supposed to be differentiated from the rest, to be indestructible, and to possess internal energy and also external energy (of translation). Vortex atoms are an example of this. This new point of view reduces all the phenomena of physics to problems in hydrodynamics, and the explanation of all mutual actions of atoms is sought in the motion existing in the medium. The constant recurrence of the same types of equations in all branches of physics thus receives a new meaning. Great light has been thrown on this view by the experiments of Bjerknes of Christiania, who showed that spherical substances oscillating in a fluid medium act like magnetic molecules with axes in the directions of oscillation, whence he gave the name of hydromagnetism to the phenomena. Neglecting rotations, the motion of an atom will consist of vibration and translation. If the vibra-

tions are of the same kind as those to which light is due, the velocity of translation due to planetary motion in space may be neglected in comparison with the rate at which a point on the surface of an atom is capable of moving. In a former paper (*Quart. Journ. Math.*, **20**, 77) the author has shown that the expression for the energy of a system of two atoms contains four significant terms. The first involves only vibrations, perhaps analogous to those which produce heat and light. The second involves only oscillations, perhaps analogous to those which produce magnetic phenomena. The third and fourth involve both vibrations and oscillations, and may possibly give rise to phenomena involving relations between light and magnetism. In the first place, the author supposes no "hydromagnetic" oscillations to exist, so that only vibrations have to be considered. For simplicity in the mathematical treatment he supposes the atoms to be spherical and the vibrations to consist of pulsations of the entire surface.

When the atoms are free, these pulsations will consist of a definite number of beats of fixed periods, and as there seems reason to believe that a gas at low pressure, and not too hot, gives only one bright line, it is probable that each atom has only one period of free vibration.

The constraint due to the approach of other atoms would ultimately give a continuous range of beats corresponding with the observed change in the spectrum of a rare gas gradually changing to a solid. A molecule may consist either of like or unlike atoms, and its pulsations may be either free or forced, but those of its constituent atoms must necessarily be forced. The free pulsations of a molecule may have no apparent relation to those of its atoms, just as the spectrum of a compound gas has no obvious relation to the spectra of its constituents, and a substance consisting of like atoms may exist in various molecular states, each with its own spectrum.

In the hydrodynamical investigation of these pulsating spherical atoms, the author assumes the existence of a velocity-potential, and, therefore, that there is no rotational motion of the ether outside the atoms. As the surface-condition, he assumes the velocity of the fluid normal to the surface of the atom to be equal to the normal velocity of the surface. He then considers the case of 2 atoms, and the resulting equation admits of solution in the ordinary manner in a series of zonal harmonics. The form of the expression thus obtained suggests the generalisation of considering, instead of simple pulsations, polar vibrations symmetrical about the line of centres. These would be the forced vibrations in one atom produced by free pulsations in the other. The kinetic energy of the fluid is next determined. To obtain the kinetic and potential energies of an atom, some hypothesis has to be made about its nature, and the author makes the assumption that it is a solid, elastic sphere. A curious result then comes out. The vibrations of the fluid are found to have the effect of increasing, by a definite amount, the quantity expressing the mass of the atom, so that if the atoms were without actual mass, they would still have an apparent mass due to the vibrations of the fluid. The same would apply to a linear motion of atoms in the ether, but in either case, in order to produce the vibrations, the atoms or their surfaces must possess potential energy. An atom might, therefore, be the one im-

material thing in the universe, consisting of a perfect vacuum (even as to ether) in the boundary of which potential energy in some manner resides.

Suppose, now, that the distance between the atoms is so great that they have no mutual action. Then we can determine the free vibrations produced by a "single pole" disturbance, if the atom is disturbed, and then left to vibrate freely in the ether. Assume the free vibration to be a pulsation, and it will correspond with the single bright-line vibration. Then it appears that if the actual mass of the atom is not zero, the potential energy resident in the surface must contain a factor determined by the mechanical structure of the atom, depending on its elasticity, for example. The period will, therefore, probably depend on the density, or on the radius, or on both.

Now, according to Lockyer (*Studies in Spectrum Analysis*, 143), "There is a noteworthy connection between the atomic weight of metalloids and the region of the spectrum where their lines appear under similar conditions." A careful examination of the position of the single bright lines and of the atomic weights of different substances might thus throw some light on the mechanical structure of atoms.

It should be noted that the natural pulsations of an atom are of greater importance than its other free vibrations, because the latter will be about different sets of poles in different atoms, so that when the atoms after collision pass beyond the sphere of mutual action, these vibrations will not have a common direction as in the case of the pulsations, so that they will produce less effect on the ether.

The free vibrations of a pair of mutually acting atoms are next considered, and it is found that a new set of periods is introduced which are functions of the distance between the atoms. If this distance is great, these periods do not differ much from that of free vibration of either. There will thus be a number of periods grouped about the natural periods. If they correspond with light vibrations, we should expect the spectrum lines to spread out into bands when the atoms of a gas are brought nearer together. When the distance becomes very small, the vibrations of any pair will differ considerably from the free vibrations of either, and, as they depend on the distance, they will vary from pair to pair, so that, ultimately, there will be a continuous spectrum. Again, if a chemical combination consisted of two atoms in close union, we should not expect the spectrum to consist of their separate spectra superposed.

In the case of three or more atoms, the consideration of polar vibration presents great difficulties, so the author here considers pulsations only. He arrives at the conclusion that a gas composed of molecules, each containing three different atoms, will, when at low pressure and not too high temperature, have a spectrum consisting in part of two bright lines, one nearer to the violet and the other nearer to the red than any bright line due to the component atoms pulsating freely. He finds that the more nearly the pulsation periods of two atoms agree, the more intense will be the vibrations of the molecule formed by their union.

For a molecule composed of three atoms, of which two only are alike, he finds that there will be two bright lines equidistant on each

side of the line due to the like pair, and one bright line farther from this line than the original line due to the unlike atom.

For a molecule consisting of three similar atoms, there will be two bright lines nearer to the violet, and one nearer to the red than that due to the atoms, and if the atoms are equidistant, the two former will coincide.

For a molecule consisting of two atoms of different periods there will be two bright lines, one nearer the violet and the other nearer the red than the originals.

For a molecule consisting of two atoms of equal periods, there will be two bright lines, the lower further towards the red than the upper one towards the violet.

In the case of three similar and equidistant atoms, the line nearer to violet will be brighter than in the case of two atoms. This suggests experiments on spectra of the same element in different molecular conditions, such as oxygen and ozone.

It is also shown that the dissociation of a diatomic molecule ought to be accompanied by a greater generation of heat, the nearer the single bright line is to the violet end of the spectrum; for example, the fundamental line of hydrogen should be nearer the violet than that of carbon.

The author then proceeds to consider a number (p) of mutually reacting atoms of different kinds, and he shows that the spectrum of the combination may be deduced when the single bright lines given by the component atoms are known.

It is shown that the sum of the squares of the periods of pulsation (whether under mutual action or free) is always constant, and as a special case, if, owing to the effect of pressure or temperature, an element gives p spectral lines instead of a single bright line, then the sum of the squares of the periods corresponding with the p bright lines is equal to p times the square of the period corresponding with the single bright line.

In calculating a theoretical spectrum, the author points out that we must not expect always to have as many bright lines as there are atoms in the combination, as some of them may fall in the invisible portions of the spectrum.

Since the periods of a combination depend on the central distances between the component atoms, a combination of atoms oscillating about a mean position would give a fluted spectrum.

Again, if there are a great number of atoms at each instant within the sphere of mutual action, there will be a great number of pulsations corresponding with all the central distances, and the complexity of these lines may make them appear like bands, and so give rise to a fluted spectrum. This would be more or less the case for an element not in a molecular state, and the atoms of which come very close together.

By considering the number of atoms within a sphere of given radius, it is easily seen that if the spectrum of an element has, say, 300 lines at the ordinary pressure, it will have not more than seven lines at 1/64 of the ordinary pressure, and before the pressure falls to 1/125 it will only give one line, supposing the temperature to remain constant, and that the ultimate atoms, supposed uniform, have only

one free pulsation. This appears suggestive in relation to the spectrum of iron. It is assumed, of course, that the ultimate atoms of the element are uniform and have only one free pulsation.

The difference between the spectrum of a mixture and of a chemical combination introduces the idea of atoms united into groups. The forces between the constituent atoms are called *chemical forces*, those between groups or molecules at small distances *molecular forces*, at great distances *gravitational forces*.

To see what light the theory throws on these three kinds of forces, the author considers the expression for the total kinetic energy of atoms and fluid, and supposes only pulsations without polar vibrations. He also assumes that the linear velocities of the atoms are not of a magnitude comparable with those supposed to produce magnetic phenomena.

Chemical Forces.—The consideration of two atoms in the same molecule, then, leads to the result that the atomic force between them must vary, partly as the inverse cube, and partly as the inverse fifth power of their central distances. For molecular union, the force must be attractive, which leads to the condition that the product of the potential energy coefficient by the intensity of free pulsation must be greater for the atom having the greater free period. When the central distance is large relatively to the atomic radii, the forces of chemical combination vary as the inverse cube. For two equal atoms the law is the same, provided the pulsations are of equal intensity; if not, the force is made up of the inverse square, cube, fourth and fifth powers of the distance, the second always repulsive, and the others attractive or repulsive, and it is found that unless a decrease in the period denotes a decrease in the atomic radius, the force of chemical combination will increase as the periods decrease, so that a molecule giving lines in the violet will be more stable than one giving lines in the red.

The author has not been able to determine whether the equilibrium in a diatomic molecule is statical or whether one atom describes an orbit about the other. Whether the former is possible except for atoms in contact depends on their internal structure, about which we know nothing. In the case of two unlike atoms, or of two like atoms having equal pulsation amplitudes, describing a relative orbit, the latter is not in general a closed curve, so that permanent union would be impossible. The author thinks it probable that the nature of the equilibrium may depend on the molecular state of a body. For example, the molecules of a rare gas may be in dynamical equilibrium, describing orbits about each other, while those of solids may be in statical equilibrium.

Consider p unlike atoms in a molecule. Let the chemical intensity be defined as the product of the potential energy coefficient into the intensity of free pulsation, and let the difference of the chemical intensities divided by the difference of the squares of the periods be called the chemical coefficient. Then it is found that the atoms will attract, and can therefore form a molecule, if this coefficient is positive, whilst if it is negative they will repel. When a_1 and a_2 are the radii of two atoms, and τ_1 and τ_2 the potential energy coefficients, the force between the two atoms is found to be—

$$\frac{\text{Chemical coefficient}}{(\text{Central distance})^3} \times \frac{64\pi^4 a_1^2 a_2^2}{\tau_1 \tau_2},$$

which the author calls—

$$\frac{\text{Chemical affinity}}{(\text{Central distance})^3}.$$

A knowledge of the chemical affinity for every pair of p atoms would enable us to determine the possibility of the p atoms uniting to form a molecule, and also the stability of the molecule if it could be formed.

Suppose a third atom to be added to a pair already in combination. Let $2\pi/\nu_1$ and $2\pi/\nu_2$ be the periods, and I_1 and I_2 the chemical intensities of the combined atoms; and let $2\pi/\nu_3$ and I_3 represent the same quantities for the third atom. Suppose ν_1 greater than ν_2, and, therefore, I_1 less than I_2. Then the following results are obtained :— Case I, ν_3 greater than ν_1.

Combination is possible if I_3 is less than I_2 or I_1, as the third atom is attracted by both the combined atoms. If I_3 is greater than either I_2 or I_1 combination is impossible, as the third atom is repelled by both. If I_3 lies between I_2 and I_1 the third atom is attracted by one and repelled by the other. Let A_{12} be the chemical affinity between the two combined atoms, A_{13} and A_{23} those between the combined and uncombined, and let γ_{12}, γ_{13}, γ_{23} be the distances between the atoms. Then if $-A_{13}/\gamma_{13}^3$ is less than A_{23}/γ_{23}^3 the third atom will remain in the combination. If A_{12}/γ_{12}^3 is less than $-A_{13}/\gamma_{13}^3$ the first atom will be turned out of the combination and the third substituted for it, forming a new stable diatomic molecule. If A_{12}/γ_{12}^3 is greater than $-A_{13}/\gamma_{13}^3$ the third atom will, as in the last case, attract the second and repel the first, but the first will remain in the combination, so that the molecule will contain two mutually repelling atoms united by a third atom attracting both of them, so that the result will be an unstable molecule. Molecules of this character give rise to unstable and explosive compounds.

Other cases in which ν_3 lies between ν_1 and ν_2, and in which it is less than either, give results of the same character. The investigations appear to indicate that it would be impossible for an element in its dissociated state, that is, with its atoms not in molecular union, to break up a diatomic compound in such a manner that all these elements should be dissociated. The law indicated in this case is easily generalised.

The author points out that the results which have been considered show that his theory would make all questions of chemical combination or decomposition one simply of calculation, if the free pulsation of every elementary atom could be observed, and chemical intensities and affinities tabulated.

Molecular Forces.—In a gas at ordinary pressure and density the atoms in each molecule may be considered to be executing forced pulsations under the influence of the other atoms of the same molecule, but independently of the atoms in other molecules. On this supposition the author shows that the force between two molecules will vary partly as the inverse square, and partly as the inverse fifth power of

their central distance. The latter will always be attractive; the former will also be attractive provided like atoms in like molecules are pulsating in the same phase, and also for certain other relations between the phases, which, however, are of too arbitrary a character to be at all probable. Now a solid always gives a gas composed of like molecules, so that the cohesion of a solid must be due to molecular and not to chemical forces, and therefore it is impossible to suppose the forces between molecules partly repulsive. Thus it seems probable that like atoms in like molecules must really be pulsating in the same phase, and this receives independent confirmation from considerations founded on the undulatory theory of light; the author, therefore, assumes it to be true.

Every term of the intensity of this attractive force contains as a factor the reciprocal of the square of some period of the molecular pulsations. Let $2\pi/n$ be the period of a molecular pulsation, a the radius of an atom, then it is reasonable to assume that a^4n^2 will increase with n, and in this case it follows that the molecular forces will be greatest for substances which give bright lines nearest to the violet, so that their force of cohesion, or toughness, when in a solid state will be greatest, or the toughness of pure metals should be in the same order as the rates of vibration corresponding with their single bright lines.

The conclusion that like atoms in like molecules must be pulsating in the same phase is found to apply also to free atoms, or atoms which are not in molecular union with other atoms, but when the free pulsation is altered to a forced one, the phase may be changed, it being then required only that the alteration should be a function of atomic radius / central distance, and should vanish with this quantity.

When a substance is under constant conditions, the chemical affinity cannot be supposed to vary greatly from molecule to molecule, so that not only the phases, but the amplitudes of the atomic pulsations of the molecules must be very much alike for the corresponding atoms of different molecules.

Although the action of a single atom on another vanishes when their distance is great in comparison with the atomic radii, nevertheless a great number of atoms may produce a considerable total effect on a single atom, even when at comparatively great distances from it, provided their effect is to produce a wave in the ether of the same, or nearly the same, period as the free period of the single atom.

The amplitude of the free pulsation, or, in the case of an atom in a molecule, the amplitude of the forced pulsation, not differing greatly in period from that of the free pulsation, will be most affected. This change of amplitude will affect the chemical intensity of the atom, and therefore the chemical affinity of each pair of atoms, which explains the physical and chemical effects of waves of different periods (chemical, light, and heat waves), as they alter the chemical intensities of the atoms, the chemical affinity between the atoms of the same molecule, and also the molecular forces acting between the different parts of the substance.

If magnetism and electricity consist in oscillations of the ultimate atoms they will not affect their pulsations directly, but they will do so indirectly by altering their distances. When, however, atoms are

pulsating freely, alterations in the atomic distances have no effect on the pulsations, so that according to the author's theory magnetism and electricity should have no effect on the spectra of completely dissociated gases.

It should be noted that the state of a substance will not be fully defined by the nature of the atoms and of the manner in which they are united to form molecules, for the condition of a molecule at any moment will be a function of the ether disturbance in its neighbourhood, so that the physical and chemical properties of a body will depend on the state of the neighbouring ether. If the exact nature of the ether disturbance in the neighbourhood of a molecule at any moment could be ascertained, every property of the molecule could be determined without reference to other molecules.

Gravitational Forces.—The motion of translation, or oscillatory motion of two molecules, gives rise to a force of magnetic character between them. Now it is difficult to imagine any effect from the rotation of a spherical molecule in a perfect fluid, and even if the molecules are ellipsoidal it is found that the effect of their rotation is not to produce a mutual action of the nature of gravitation. The explanation of gravitation must therefore be sought in the pulsations.

The author finds that if the law of gravitation holds for all atoms, then every atom must have at least one pulsation of common period and phase with every other. He also arrives at the conclusion that all the atoms in the universe must have begun to pulsate at the same moment. He finds that the force of gravitation is not exactly mass/(distance)^2, but a certain function of the constitution of the two bodies divided by the square of the distance. In solid substances this may be almost exactly equal to the product of their masses, but in some cases it may vanish, as for instance in the case of two gases vibrating freely and having no common period of pulsation. This would not open the possibility of a substance being without weight, for since all solids and liquids give continuous spectra they must have pulsation periods in common with one or more of the periods of every other substance. It would, however, lead to the result that the density of a substance if measured by the attraction exerted between it and another substance, would vary to some extent according to the substance selected as a standard of comparison. As a matter of fact very different results have been obtained by different methods for the mean density of the earth, the values ranging from less than 5 up to 6·6, and even in Baily's experiments the average mean density obtained from experiments with balls of one substance on the torsion rod, differed from the results obtained when balls of another material were used.

The author shows that change in the atomic conditions of a substance need not affect the weight. In the cases which he considers it is found that the conditions for the weight to remain unaltered lead to relations previously proved to be true. The theory, however, leads to the result that the weight of a substance must be increased, though the increase may be very minute, by absorption of light or heat, as the effect of this will be to increase the amplitude of the pulsations. These discrepancies with what are considered well established physical facts may indicate that the pulsations are insufficient alone to explain gravitation, and that this may depend also on the shape of the atoms,

and on the nature of their vibration, or more probably on the nature of the medium in which they are vibrating. If, for example, the ether is not a perfect fluid, there may be something of the nature of skin-friction between the atoms and the ether, and in that case it is possible that gravitation may be due to rotation of the atoms.

The theory discussed in this paper is certainly one of great interest and suggestiveness, and appears to throw great light on many problems lying on the borderland of chemistry and physics. The few discrepancies which have been noted are not surprising, as practically nothing is known about the nature of the ether. Some of the phenomena of light appear to lead to the conclusion that it is a perfect fluid, as assumed in the preceding paper, whilst others appear to require it to have the properties of a very elastic solid.

G. W. T.

Note.—Sir William Thomson has pointed out (*Baltimore Lectures on Molecular Dynamics*) that there is no contradiction involved in supposing the ether to act like a perfectly elastic solid for vibrations executed in the 100 or 1600 billionth of a second, and yet to be highly mobile to substances moving at a rate of, say, 20 miles a second, and points out as an illustration that a tuning-fork can be made of Burgundy pitch when the period is a small fraction of a second, yet a bullet will, by its own weight, penetrate several inches into it in six months.

It should be borne in mind that, as stated on page 903, the spherical form of the atoms is assumed simply to facilitate the mathematical analysis, and the author, in a letter to the abstractor, points out that if the atoms are of any other shape, provided they are capable of changing their external form, and especially their size, their treatment as pulsating spheres will probably give a first approximation to the true theory. G. W. T.

Lecture Experiment. By B. F. Oettel (*Chem. Zeit.*, **11**, 1601).—A small heap of finely powdered potassium chlorate supported on a piece of filter-paper on a tripod is saturated with a solution of phosphorus in carbon bisulphide. As soon as the latter evaporates, the mass explodes with loud report and clouds of smoke. D. A. L.

Inorganic Chemistry.

Relative Values of Atomic Weights of Hydrogen and Oxygen. By J. P. Cooke and T. W. Richards (*Amer. Chem. J.*, **10**, 191—196).—It was suggested by Rayleigh (this vol., p. 643) that the balloon used for weighing hydrogen (this vol., p. 647) suffered compression when exhausted of air; the air buoyancy was thus unknowingly diminished, and the apparent weight of hydrogen thereby reduced below its true value. The shrinkage was determined by weighing the globe under water after it had been nearly filled with water and then exhausted at the pump, and again after its vacuous space had been filled with air. The contraction due to exhaustion amounted to about 1·6 c.c., and the consequent average correction to be added to the amount of hydrogen is 0·00198 gram, whence the corrected atomic

weight of oxygen = 15·869, and the composition of water is oxygen (Dumas) 88·864 ± 0·0044, and hydrogen (Cooke and Richards) 11·193 ± 0·0011, total 100·057. There is therefore in Dumas' or in the authors' method some constant source of error to account for the excess of 0·057 per cent. That the hydrogen was not perfectly burnt, as suggested by Rayleigh, seems, for several reasons given, improbable; the balance and weights were verified, and an allowance was made for the air dissolved in the water weighed.

The correction now made for the shrinkage of the glass balloon by decreased pressure has not before been made, and it obviously must be applied to all determinations of gas or vapour-densities hitherto made, and to all atomic weight determinations of any kind which involve the calculation of the weight of a measured volume of any gas or vapour; except, however, in the case of hydrogen the correction will be inconsiderable. H. B.

Potassium Hypoiodite. By C. M. van Deventer and H. L. van't Hoff (*Chem. Centr.*, 1888, 362—363, from *Maandbl. Naturwetensch.*, **14**, 98).—That potassium hypoiodite is contained in a solution of iodine in potassium hydroxide, provided the solution is made in the cold, is shown by the fact that it behaves quite differently to one which has been heated and then allowed to cool. When to a cold solution of iodine in potash, alcohol is added, iodoform is formed in large quantity, whilst in a cold solution, which has previously been heated, the brown colour has disappeared, and alcohol does not generate any iodoform. Another proof is contained in the fact that a far greater quantity of iodoform is formed when iodine is added to a solution of potassium hydroxide in alcohol than when alcohol is added to a solution of iodine in potassium hydroxide. In the latter case, the hypoiodite is partly converted into iodate, whilst in the former the potassium hypoiodite can react with the alcohol when first it is formed.

J. W. L.

Colloidal State of Sulphides. By C. Winssinger (*Bull. Soc. Chim.*, **49**, 452—457).—By employing one of the following methods, all the sulphides which were examined, except manganese sulphide, were obtained in the colloidal state. (Compare Spring and de Bock, this vol., pp. 227, 228). (1.) The precipitated sulphide is washed by decantation with pure cold water or a dilute solution of hydrogen sulphide; as the impurities are removed, the sulphide gradually becomes soluble and finally dissolves completely. (2.) The sulphide is formed in a solution so dilute that all extraneous compounds can be removed by dialysis before the sulphide becomes insoluble. (3.) The sulphide is formed in a liquid without the introduction of any substance capable of causing coagulation.

Mercuric sulphide, prepared by precipitating a dilute solution of a mercuric salt with hydrogen sulphide, was treated by the first method. The concentrated solution thus obtained is black and opaque, but a dilute solution is brown with a slightly greenish tint by reflected light. A sufficiently dilute solution can be boiled until the whole of the hydrogen sulphide is expelled, or it can be kept for more than a month, without undergoing any alteration. Colloidal zinc sulphide

is easily obtained by the first method, or by passing hydrogen sulphide through water containing pure zinc hydroxide in suspension. The solution is slightly opalescent, and can be boiled until the whole of the hydrogen sulphide is expelled without undergoing change; when boiled for a longer time the sulphide is precipitated, but, after cooling, it is readily redissolved when a stream of hydrogen sulphide is passed through the solution. A dark-brown solution of tungsten trisulphide is obtained when a little more dilute hydrochloric acid than is necessary to saturate the alkali is added to a solution of sodium thiotungstate. The solution can be filtered through paper.

A solution of molybdenum sulphide, prepared by adding an excess of dilute acetic acid to a solution of potassium thiomolybdate and dialysing, coagulates when filtered through paper. Colloidal indium sulphide is obtained when a stream of hydrogen sulphide is passed through an aqueous solution of the hydroxide; the dark golden-yellow solution can be freed from hydrogen sulphide by boiling, but it is coagulated by acetic acid or salts.

The sulphides of thallium, lead, bismuth, platinum, gold, palladium, and silver were prepared in the colloidal state by the second method; they all form reddish-brown solutions. Solutions of the sulphides of gold and silver can be kept for more than two months without change. Very dilute solutions of the sulphides of iron, nickel and cobalt were obtained by the second method; they are greenish-brown, oxidise very readily, and are very prone to coagulation.

All the solutions, with the exception of that of zinc, give very similar absorption-spectra; a dark band extends from the extremity of the violet to the green, where it gradually disappears; the extreme end of the red is faintly obscured, and the whole visible spectrum is only very slightly luminous. The spectra of the sulphides of indium and cadmium (compare Prost, this vol., p. 653) can be readily distinguished; they are more luminous, and the dark band ceases more abruptly in the region of the green.

The facility with which the sulphide of an element can be obtained and retained in the colloidal state seems to be a function of the atomic weight of the element. F. S. K.

Sodium Dithiopersulphate. By A. Villiers (*Compt. rend.*, **106**, 1354—1356).—In the preparation of this salt (this vol., p. 649) it is not necessary to cool the solution with ice. The crystals may be separated from admixed trithionate by exposing the mixture to air. The trithionate effloresces and the unaltered crystals of the dithiopersulphate are easily picked out. Tetrathionate is produced in addition to the other two salts, and it is possible that the reactions take place in accordance with the equations: $2Na_2S_2O_3 + 3SO_3 = Na_2S_4O_6 + Na_2S_3O_6$, and $2Na_2S_2O_3 + 8SO_2 = Na_2S_4O_6 + Na_2S_4O_8$.

Sodium dithiopersulphate crystallises in anhydrous, rhombic prisms with the faces *m* terminated by acute bevels formed by the faces *e*, the angles being *mm* 130°·2; e_1e_1 86°·10; e_1m 108°. When the salt crystallises at a low temperature, it combines with 2 mols. H_2O. The anhydrous crystals do not alter when exposed to air, but melt at about 125° and intumesce at 140°, giving off sulphurous anhydride and

leaving a residue which contains a sulphate and free sulphur, but no alkaline sulphide. The hydrated salt decomposes slowly in the cold and rapidly when heated, with evolution of sulphurous anhydride and formation of the trithionate. Sulphurous anhydride has no action on the trithionate at the ordinary temperature.

The dithiopersulphate does not reduce potassium permanganate even when heated, but is converted into sulphate by the action of bromine, five equivalents of bromine being required for each equivalent of the salt, a result which confirms the formula ascribed to the salt. Its solution gives no precipitates with metallic salts except mercuric chloride, which produces a precipitate of sulphur. When boiled with cupric sulphate, there is no precipitate of cuprous sulphide as with the trithionate, except after prolonged ebullition; concentrated mineral acids seem to liberate dithiopersulphuric acid from its salts without decomposing it. Nitric acid above a certain strength, however, oxidises it rapidly, and even explosively, with precipitation of sulphur and evolution of nitrogen oxides.

The name dithiopersulphuric acid must be regarded as merely provisional. C. H. B.

Preparation of Nitrogen Tetroxide. By B. Setlick (*Chem. Centr.*, 1888, 461, from *Listy Chem.*, **11**, 241—242).—The technical application of liquid nitrogen tetroxide to the preparation of explosive substances led the author to determine the yield of tetroxide obtained by the usual methods of preparation. 60 to 70 per cent. of the theoretical yield was obtained by heating lead or calcium nitrate, and by reducing nitric acid with starch, &c. The author has also investigated the yield of nitrogen tetroxide on oxidising nitric oxide with pure oxygen and air. The nitrogen tetroxide when formed was led through two cooled receivers to condense the gas, and then through sulphuric acid to dissolve the remainder. With air, the yield was 138·6 grams (of which 18 grams condensed); with a mixture of equal parts of air and oxygen, 129·8 grams (of which 80·3 grams condensed) out of a theoretical yield of 156·8 grams. With oxygen alone, the yield was 92·8 grams (of which 74 condensed) out of a possible 102 grams. J. P. L.

Preparation of the Alkali Salts of Hydroxylaminedisulphonic Acid and of Hydroxylamine. By F. Raschig (*Chem. Zeit.*, **12**, 219).—Alkaline hydroxylaminedisulphonates may be prepared by adding the alkaline hydrogen sulphite (2 mols.) in concentrated aqueous solution to the alkaline nitrite (1 mol.) at a temperature not exceeding 0° for the sodium salt or 40° for the potassium compound. They may also be prepared by passing sulphurous anhydride to excess into a solution containing the alkaline nitrite (2 mols.) and either the alkaline carbonate (1 mol.), or the hydroxide (2 mols.). The potassium salt may also be obtained by adding a soluble potassium salt to a solution of sodium hydroxylaminedisulphonate, in which case the potassium hydroxylaminedisulphonate settles out as a compact, crystalline mass. When solutions (which must not react alkaline) of these salts or of the hydroxylaminemonosulphonates are heated for

some time at 100°, or for a short time at 130°, they are resolved into hydroxylamine and alkaline sulphates, which may be readily separated by fractional crystallisation. D. A. L.

Electrolysis of the Acids of Phosphorus. By G. Janeček (*Chem. Centr.*, 1888, 273—274, from *Rad jngoslavenske Akad.*; Agram, **83**, 186—200).—The electrolysis of melted orthophosphoric acid at a temperature below that at which it becomes dehydrated, dehydrates it rapidly, hydrogen and oxygen being evolved at the electrodes. The dehydration proceeds first to the formation of pyrophosphoric acid, and later to that of metaphosphoric acid. The liberated oxygen contains a quantity of ozone. The metaphosphoric acid is further decomposed with formation of phosphorous acid, large quantities of spontaneously combustible phosphoretted hydrogen being evolved at the same time. J. W. L.

Pyrophosphorous Acid. By L. Amat (*Compt. rend.*, **106**, 1400—1402).—When the salt $2NaH_2PO_3 + 5H_2O$ is heated at 160°, it loses 6 mols. H_2O, and yields the pyrophosphite $Na_2H_2P_2O_5$. The crystals should previously have been partially dehydrated in a vacuum. Sodium pyrophosphite is very soluble in water, and the solution gives no precipitate with silver nitrate, but metallic silver separates out very slowly in the cold, more rapidly on heating. Monosodium phosphite gives with silver nitrate an immediate white precipitate which rapidly becomes black. The pyrophosphite is neutral to methyl-orange and phenolphthaleïn, whilst the phosphite is neutral to methyl-orange but acid to the phthaleïn. A cold solution of the salt alters very slowly, but if heated, it is reconverted into the phosphite. If the boiling solution is gradually mixed with sodium hydroxide, disodium phosphite is obtained.

Only the monosodium salts of the monobasic acids, such as metaphosphoric and hypophosphoric, are neutral to phenolphthaleïn, and since the sodium salt of pyrophosphorous acid is neutral to the phthaleïn, it follows that this acid is also monobasic. C. H. B.

Note by Abstractor.—The conclusion that pyrophosphorous acid is monobasic is only tenable on the old notation, O = 8. Adopting the modern notation, it must be at least bibasic. C. H. B.

Arsenic Compounds. By K. Preis (*Chem. Centr.*, 1888, 657, from *Listy Chem.*, **12**, 141—148).—*Sodium Oxythioarsenates.*—The reaction between arsenic trioxide and a solution of sodium sulphide varies according to the respective masses employed. The compounds described below are formed by the interaction of 1 molecular proportion of arsenic trioxide with 2 of sodium sulphide. In the warm, a brown precipitate is produced; if the turbid solution is boiled for five minutes, filtered hot, and then allowed to cool, it is converted into a magma of colourless needles from which four oxythioarsenates, besides normal sodium arsenate, were obtained by fractional crystallisation: (1) $Na_3AsSO_3 + 12H_2O$; (2) $Na_2HAsSO_3 + 8H_2O$; (3) $Na_3AsS_2O_2 + 2H_2O$; (4) $Na_{12}As_4S_5O_{11} + 48H_2O$. J. P. L.

Crystalline Silicic Acid. By H. Hager (*Chem. Centr.*, 1888, 461, from *Pharm. Centralb.*, **29**, 115).—To prepare this crystalline silicic acid, the author treats siliceous limestone with crude concentrated hydrochloric acid until carbonic anhydride ceases to be evolved, then filters and adds to the filtrate 29 to 31 per cent. hydrochloric acid until a cloudiness appears. After remaining 24 hours the solution is decanted, the precipitate strained and washed, finally with distilled water, then dried on porcelain. Silicic acid so obtained is crystalline and corresponds to the formula $H_2SiO_3 + 3H_2O$ or $H_2SiO_3 + 2H_2O$. It is more soluble than the amorphous variety, and probably preferable as an indifferent covering for wounds and for administering internally. J. P. L.

Alkaline Phosphites. By L. Amat (*Compt. rend.*, **106**, 1351—1354).—*Monosodium phosphite*, $2NaH_2PO_3 + 5H_2O$, is obtained by adding sodium hydroxide or carbonate to phosphorous acid until the solution is neutral to methyl-orange. The liquid is then concentrated by evaporation and the salt crystallises. Not unfrequently a supersaturated solution is obtained which crystallises in contact with a fragment of the solid phosphite. If no crystals of the phosphite are at hand, the solution is concentrated until it has the composition of the crystals and is cooled to a low temperature. The salt is recrystallised from water and dried by exposure to air. It can also be obtained by the action of phosphorus trichloride on sodium carbonate, but the separation of the sodium chloride is difficult.

Monosodium phosphite is very soluble in water and melts at 42°. At 100° the fused salt loses water and the liquid deposits crystals which may be either a lower hydrate or the anhydrous salt. If heated at 100° for a long time, it becomes completely dehydrated; it also becomes anhydrous in a dry vacuum. When heated above 130° the phosphite changes into sodium pyrophosphite.

Potassium phosphite is obtained in a similar manner. It forms anhydrous crystals, which are difficult to purify by recrystallisation. C. H. B.

Sodium Trithionate. By A. Villiers (*Compt. rend.*, **106**, 1356—1357).—Sodium trithionate, $Na_2S_3O_6 + 3H_2O$, crystallises in rhombic prisms with the faces m and g terminated by the domes e_1e_1. The angles are as follows: mm 126° 30′; mg_1 116° 40′; e_1g_1 124° 50′; e_1m 105° 10′; e_1e_1 110° 14′. C. H. B.

Extraction of Lead from Residues obtained in the Manufacture of Zinc. By E. Prost (*Bull. Soc. Chim.*, **49**, 682—685).—The residues experimented on are obtained in the reduction of zinc and are called "lead-cinders;" they are separated mechanically into "fine cinders," containing 15 to 20 or even 22 per cent. of lead, and "granulated metal" containing at the most 10 per cent. of lead. The richer samples contain as much as 400 grams of silver per tonne and the "granulated metal" 225 grams per tonne. The following analyses show the average composition of these residues:—

Fine Cinders.

	(1).	(2).
Pb	18·25	17·22
SiO_2	15·52	16·00
Fe	36·50	38·10
Zn	6·14	5·00
CaO	5·40	6·15
Carbon (approx.)	3·00	3·00
Ag per tonne	340·0 grams	340·0 grams.

When the "fine cinders" are placed in a crucible, covered with a layer of powdered coal and heated in a wind-furnace, the whole of the lead excepting the portion which volatilises is obtained in the metallic state. A mixture of equal parts of "fine cinder" and "granulated metal," containing 12·57 per cent. of lead, gives a readily fusible slag, the composition of which is SiO_2 50·85 per cent., Al_2O_3 5·10 per cent., FeO 20·14 per cent., and CaO 23·54 per cent. A similar mixture containing 13·06 per cent. of lead is more refractory, but when heated in very thin layers (of about 2 cm. thickness) fusion is complete, and about 84 per cent. of the lead is recovered. The composition of this slag is SiO_2 50·84 per cent., FeO 17·09 per cent., Al_2O_3 11·54 per cent., CaO 19·96 per cent. Fusion takes place most rapidly when the silica, lime, and alumina are in the proportion shown by the first analysis.

The lead contains almost the whole of the silver; in cases where the yield of lead attained 84 per cent., it contained 92 per cent. of the silver present in the original mixture. F. S. K.

New Halogen Compounds of Lead. By R. G. Grisson and B. Thorp (*Amer. Chem. J.*, **10**, 229—232).—*Lead chlorothiocyanate*, $PbCl_2,Pb(CNS)_2$, is obtained by digesting the chloride with potassium thiocyanate and then recrystallising from boiling water. Ammonia converts it very slowly into lead hydroxide, and neither of the acid radicles is removed before the other. *Lead bromothiocyanate*, $PbBr_2,Pb(CNS)_2$, similarly produced, forms slightly brownish crystals, the salt, $8PbBr_2,Pb(CNS)_2$, is obtained by acidifying with hydrobromic acid. *Lead iodothiocyanate*, $PbI_2,3Pb(CNS)_2$, is formed with some little difficulty.

Lead chlorocyanide, $PbCl_2,2Pb(CN)_2$, is formed by digesting lead chloride with potassium cyanide; it is quite insoluble in water, and decomposes on keeping. No double salts of the ferrocyanide could be obtained.

Lead Bromiodide.—By crystallising a solution of lead iodide in hydrobromic acid, the following substances were obtained: $PbBr_2,PbI_2$; $3PbBr_2,PbI_2$; $6PbBr_2,PbI_2$; and lastly lead bromide. *Lead chlorobromiodide* was prepared by crystallising a solution of all three salts: the crystals contain uniformly 61·5 per cent. of lead. H. B.

Relation of the Atomic Weights of Copper and Silver. By T. W. Richards (*Amer. Chem. J.*, **10**, 182—187).—The results recently obtained by W. N. Shaw (Abstr., 1887, 444) by the electrolytic method, are higher than those previously obtained for the atomic

weight of copper, with the exception of the determinations of Dumas; namely, Berzelius, 63·153 (O = 15·963); Erdmann and Marchand, 63·316; Millon and Commaille 62·979; Dumas (sulphide as well as oxide), 63·5; Hampe, 63·197 and 63·173; Shaw, 63·33 or 63·36. The present results were obtained by a method tried and discarded by Hampe, namely, the precipitation of silver nitrate by metallic copper. The precipitation, however, must be effected at −1°, and the solution of silver nitrate should be tolerably strong (5 to 20 per cent.). Under these conditions, the precipitated silver is very easily manipulated and is quite free from copper, whilst at higher temperatures, the action is more rapid, but gases are evolved and the silver precipitate contains copper compounds. The number Cu = 63·437 (Ag = 107·675), is obtained as the mean of six very concordant determinations, the greatest difference between the estimations being 0·020 and the probable error ± 0·0023. This number agrees nearly with that obtained by Dumas with the sulphide of copper. The method is proposed as a means of preparing chemically pure silver.

H. B.

Atomic Weight of Copper. By T. W. Richards (*Amer. Chem. J.*, **10**, 187—191).—After the completion of the previous experiments it was found that a temperature of 150°, at which this silver was dried, was not sufficient to drive off all moisture; in the only case in which this error was examined the correction amounted to +0·015. The secondary reaction, occurring at temperatures above 0° and which causes the precipitation of green basic copper nitrate, is written $4AgNO_3 + 4Cu + 3H_2O = Cu(NO_3)_2, 3Cu(OH)_2 + 2Ag_2 + NO + NO_2$. The copper of which two samples were taken (Lake Superior and German, which universally contain comparatively large amounts of arsenic) was purified much as before. The average results obtained were respectively 63·449 with a probable error of ±0·0010, and 63·450 with a probable error of ±0·0013, whilst the average result obtained from the weight of the precipitate dried at 150° is 63·436. The agreement of results points to the purity of the materials, and it is believed to the absence of any constant error. It was found impracticable to use sulphate of silver in place of the nitrate.

H. B.

Formation of Crystallised Ferric Hydroxides in the Dry Way. By G. Rousseau and J. Bernheim (*Compt. rend.*, **106**, 1530—1532).—2 grams of precipitated ferric hydroxide dried at 100° is added to 15 grams of fused sodium hydroxide in a platinum crucible, and the mixture is heated at the highest temperature of the bunsen flame for about an hour and a half, care being taken that all the sodium hydroxide is not volatilised. The interior of the cooled crucible is covered with lustrous needles and lamellæ of the composition $20Fe_2O_3,32H_2O,3Na_2O$. They dissolve readily in acids, and begin to lose water at 120. At 150°, 9·54 per cent. of water is given off, at 250—300°, 3·7 per cent., and the remainder at dull redness. The dehydrated substance resembles specular hæmatite in appearance, and its constitution seems to have been profoundly altered. It is attacked with difficulty by acids, but imparts an alkaline reaction to water. This latter fact indicates that the com-

pound has dissociated, and that the presence of water is essential to the stability of the complex molecule. Mallard has shown that ferric oxide is monobasic, and will not accumulate in a saline molecule in the same manner as silica, titanium oxide, &c. It may be assumed, therefore, that in the product described, sodium oxide has replaced water, molecule for molecule. At higher temperatures the compound loses water and sodium oxide and is converted into compounds corresponding with the lower hydrates.

When ferric hydroxide is fused with sodium hydroxide at about 110° violet-red lamellæ are formed, which contain Fe_2O_3 88·7, Na_2O 2·47, H_2O 8·61 per cent. If it is assumed that the sodium oxide has replaced water, this product corresponds with goethite, Fe_2O_3,H_2O. It loses 3·65 per cent. at 150°, 3·33 at 300°, and the remainder at a red heat.

If 2 grams of dried ferric hydroxide is heated to bright redness for half an hour with 6 grams of sodium carbonate, and the product is mixed with excess of sodium chloride and heated at an orange-red heat until the greater part of the sodium chloride is volatilised, black crystals and lustrous black plates are obtained which have the composition Fe_2O_3 83·97, Na_2O 2·81, H_2O 12·39 per cent. Assuming the soda to have replaced water, these numbers correspond with $5Fe_2O_3,7H_2O$.

If ferrous sulphate and sodium chloride are heated to redness for one hour and the product poured on a metal plate and extracted with water, crystalline plates are obtained of the composition Fe_2O_3 94·30 Na_2O 0·78, H_2O 4·09 per cent., which corresponds with turgite, $2Fe_2O_3,H_2O$. This product is less soluble in acids than those previously described; it loses 0·92 per cent. at 150°, 1·23 per cent. at 300, and the remainder at a red heat. The residue has an alkaline reaction.

All these compounds contain sodium oxide and water in the proportion of 1 mol. of the former to 10 mols. of the latter, a fact which favours the supposition that the first compound is gradually dissociated, the sodium oxide and water being eliminated simultaneously in these proportions.

No similar results were obtained with potassium hydroxide.

C. H. B.

Uranates of Ammonium and of certain Amines. By A. J. Carson and T. H. Norton (*Amer. Chem. J.*, **10**, 219—221).—The analyses of normal uranates of the metallic bases always show considerable differences between the practical and theoretical percentages of uranium. The same is now shown to hold for the uranates of ammonium, methyl-, dimethyl-, trimethyl-, ethyl-, diethyl-, triethyl-, and isobutyl-, and phenyl-ammonium. The only salt that at all agreed with the theory was the ethylammonium salt. H. B.

Influence of Hydrochloric Acid on the Solubility of Stannous Chloride. By Engel (*Compt. rend.*, **106**, 1398—1400).—The addition of hydrochloric acid to a solution of stannous chloride at first precipitates a molecule of the salt for each molecule of acid added, a result which agrees with the author's general law. As the

proportion of hydrochloric acid increases, the precipitation of the salt gradually ceases. The solubility of the stannous chloride, then, increases with the amount of acid added, but when a certain proportion is reached it remains constant.

If dry hydrogen chloride is passed over hydrated stannous chloride, $SnCl_2 + 2H_2O$, a liquid is formed which contains 19·6 per cent. of water; sp. gr. 2·199. Crystals of the hydrate, $SnCl_2 + H_2O$, are formed at the same time. The liquid product has a composition which agrees fairly well with the formula $SnCl_2,HCl + 3H_2O$. It does not crystallise at −21°, but at −40°; it deposits slender, silky needles which melt at −28°. Portions of the liquid removed at different stages of the crystallisation have the same composition as the original liquid, and it follows that the crystals are a hydrochloride of stannous chloride of the composition $SnCl_2,HCl + 3H_2O$.

C. H. B.

Action of Chlorine on Gold. By L. Lindet (*Bull. Soc. Chim.*, **49**, 450—451).—Auric chloride is formed when gold sponge is treated with chlorine at any temperature, but the product always contains a considerable proportion of unattacked metal, and, when the temperature is above 175°, aurous chloride is also present (compare Krüss and Schmidt, this vol., p. 28). The quantity of auric chloride formed increases with the temperature, but the proportion of aurous chloride increases equally.

F. S. K.

Rhodium Sesquisulphide. By E. Leidié (*Compt. rend.*, **106**, 1533—1536).—The only method which yields pure rhodium sesquisulphide, Rh_2S_3, is to heat the chloride, Rh_2Cl_6, prepared at 440°, in a current of pure and dry hydrogen sulphide at 360°. It forms blackish, crystalline plates which retain the form of the chloride. At a dull red heat in an atmosphere of nitrogen, it loses 8 per cent. of sulphur, and the residue corresponds with Berzelius's supposed monosulphide. If heated at bright redness in a vacuum or in a brasqued crucible it yields a regulus which has a metallic appearance but contains 9 per cent. of sulphur. The whole of the sulphur can only be expelled in the oxyhydrogen flame. Rhodium sesquisulphide is insoluble in alkalis and is not attacked by bromine-water nor by hydrochloric acid, nitric acid or aqua regia.

Hydrated rhodium sulphide obtained by the ordinary methods and dried contains more sulphur than corresponds with the sesquisulphide. When heated at 440° in a vacuum, it loses sulphurous anhydride and water, and the residue approximates closely in composition to the sesquisulphide. If a solution of a rhodium salt is mixed with a large excess of hydrogen sulphide in a closed vessel, precipitation of the rhodium is complete in a few minutes at 100° but requires months at the ordinary temperature. In both cases the product is a brownish-black precipitate of the hydrosulphide, $Rh_2S_3,3H_2S$, which is insoluble in alkalis and in hydrochloric or nitric acid, but is attacked by bromine-water and aqua regia. When heated with 500 times its weight of water in a closed vessel or in an atmosphere of nitrogen, the hydrosulphide is decomposed into rhodium sulphide and hydrogen sulphide. Whenever a rhodium salt

is precipitated by hydrogen sulphide at 100°, the hydrosulphide is formed, but it immediately decomposes, the change taking place more readily in presence of an excess of rhodium salt than in presence of water alone.

If a normal salt of rhodium, such as an alkaline rhodiochloride, is treated with an excess of a concentrated solution of an alkaline monosulphide, a compound of rhodium sulphide with the alkaline sulphide is precipitated, and is stable in presence of excess of the latter, but is decomposed by water. When the liquid contains not more than 16 mols. of water to each molecule of sodium sulphide the precipitate has the composition $Rh_2S_3,3Na_2S$; with at least 110 mols. of water, the precipitate is rhodium sulphide alone, and between these limits the double sulphide is gradually dissociated as the proportion of water increases. With 550 mols. of water to each molecule of sodium sulphide, the precipitate formed in the cold is yellow rhodium hydroxide, the alkaline sulphide being completely dissociated by the water into the hydroxide and hydrogen sulphide.

Potassium sulphide gives precisely similar results. C. H. B.

Rutheniates and Per-rutheniates. By H. Debray and A. Joly (*Compt. rend.*, **106**, 1494—1500).—60 grams of potassium hydroxide is dissolved in 250 grams of water, heated to 60°, and gradually mixed with 50 grams of ruthenium peroxide fused under water. Oxygen is evolved and the liquid first becomes green and then almost black. When the change is complete the liquid is allowed to cool in a closed vessel. It then deposits small crystals which are at once dried on porcelain and afterwards in a dry vacuum. When dry it does not alter on exposure to air.

Potassium per-rutheniate, $KRuO_4$, obtained in this manner, forms opaque, black, highly lustrous, quadratic octahedra, the dominant face being a', but $b\frac{1}{2}$ is also present; plane of cleavage a';

$$h : b : : 1{\cdot}15543 : 1.$$

In composition it is identical with the crystals obtained by the action of chlorine on the rutheniates. At 400° in a vacuum the per-rutheniate rapidly decomposes with evolution of oxygen and formation of the rutheniate and ruthenium dioxide, or possibly a compound of potassium oxide with a lower oxide of ruthenium. Dry chlorine decomposes the per-rutheniate in the cold with formation of potassium chloride and ruthenium peroxide.

Potassium rutheniate, K_2RuO_4, is obtained by evaporating the mother-liquor from the per-rutheniate in a dry vacuum, and also directly by adding 50 grams of the peroxide to a larger proportion of alkali (70 grams) and water (500 grams) than in the preparation of the per-rutheniate, care being taken that the temperature of the liquid does not exceed 60°. It forms rhombic prisms with the faces m,h', g^3, p, a', and a^4; $a : b : c = 0{\cdot}7935 : 1 : 1{\cdot}1973$. Measurements of the angles are given. The crystals alter rapidly on exposure to air, and even after being dried absorb moisture and carbonic anhydride. They contain 1 mol. H_2O, which is not given off at 106° in a vacuum. At 200° they become anhydrous without being reduced,

and the anhydrous salt does not alter in a vacuum at 440°. The rutheniate is very soluble in water, forming a very deep-orange solution, which, when diluted or mixed with acids, changes, in a manner similar to potassium manganate, into the per-rutheniate and the pentoxide, thus: $4K_2RuO_4 + 3H_2O = 2KRuO_4 + Ru_2O_5 + 6KHO$. With carbonic anhydride or chlorine, concentrated solutions yield a precipitate of the per-rutheniate, which decomposes into the pentoxide or the peroxide. Concentrated hydrochloric acid precipitates a mixture of the per-rutheniate and the pentoxide, but the precipitate redissolves with evolution of chlorine and formation of the sesquichloride.

When a solution of potassium per-rutheniate is mixed with an excess of the hydroxide it is converted into the rutheniate with evolution of oxygen. The decompositions of the rutheniate and per-rutheniate can be traced by the changes in colour.

Sodium per-rutheniate is obtained in a similar manner to the potassium salt, and crystallises with 2 mols. H_2O. The rutheniate does not crystallise well, and cannot readily be purified.

Barium hydroxide solution dissolves ruthenium peroxide with evolution of oxygen and formation of a black precipitate which rapidly changes into a vermillion-red, crystalline precipitate of *barium rutheniate*, $BaRuO_4 + H_2O$. The same compound is obtained by adding barium chloride to a solution of potassium rutheniate, and the strontium salt is formed as a brilliant, red precipitate in a similar manner. The calcium and magnesium salts are black.

Silver rutheniate is obtained as a very dense, black precipitate when silver nitrate is added to a solution of an alkaline rutheniate. The precipitation of the ruthenium is complete.

The per-rutheniates of the alkaline earths and the heavy metals have only a momentary existence. If barium chloride or silver nitrate is added to a solution of an alkaline per-rutheniate a precipitate of the corresponding rutheniate is formed, and the liquid has a yellow colour and contains per-ruthenic acid.

Although in their relations and decompositions ruthenic and per-ruthenic acids are analogous to manganic and permanganic acids, the rutheniates and per-rutheniates are not isomorphous with the manganates and permanganates respectively, and do not crystallise with the same quantities of water of crystallisation. C. H. B.

Atomic Weight of Osmium. By K. Seubert (*Ber.*, **21**, 1839—1847).—The atomic weight of osmium was found to be 191·12 as the mean of several analyses of two different samples of ammonium osmiochloride and potassium osmiochloride. In the author's opinion this result is rather too high, and the true atomic weight is more probably 190·8. Osmium therefore falls into its right place below iridium in the natural system.

The ammonium osmiochloride, $(NH_4)_2OsCl_6$, was obtained by adding a solution of ammonium chloride to an alcoholic solution of osmium chloride; the precipitate is washed with ice-cold water until free from excess of ammonium chloride, dissolved in water at about 50°, and filtered. When the solution is evaporated in a partial vacuum, well-defined octohedra are deposited which appear dark red in the

liquid but black when dry. The powdered substance is bright red.

Potassium osmiochloride, K_2OsCl_6, was prepared by heating the metal, obtained from the ammonium salt, with potassium chloride in a stream of chlorine, washing the product with ice-cold water until free from excess of potassium chloride, and recrystallising from lukewarm water. It is more stable in solution than the ammonium salt, to which, however, in the dry state it is very similar.

The second sample of potassium osmiochloride was prepared in like manner from a different specimen of osmium. The second sample of ammonium osmiochloride was obtained by reducing the potassium osmiochloride just mentioned in a stream of hydrogen, washing the residue until free from potassium chloride, and heating it with pure sodium chloride in a stream of chlorine. The concentrated, filtered solution of the resulting sodium osmiochloride was precipitated with a saturated solution of ammonium chloride, the precipitate washed consecutively with dilute ammonium chloride solution and ice-cold water and recrystallised from water.

The method of analysis was practically the same as that described in the case of platinum (Abstr., 1881, 514), but in weighing the finely divided osmium care must be taken on account of the readiness with which it oxidises, a fact which makes the results of doubtful trustworthiness. F. S. K.

Mineralogical Chemistry.

Minerals from New Localities in Bohemia. By F. Katzer (*Chem. Centr.*, 1888, 493, from *Tschermak's Mitth.*, **9**, 404—415).—*Bornite* (purple copper ore) from Woderad. Hardness above 3. Sp. gr. 4·91. Its composition is—

S.	Cu.	Fe.	Insol.	Total.
23·76	59·85	15·62	1·23 =	100·46

and corresponds to the formula $3Cu_2S,CuS,2FeS$.

Rutile from Sobeslau. Rutile is of general occurrence in Bohemia, especially in the neighbourhood of the Bohemian forest gneiss and the Bohemian-Moravian primitive rocks, and also in the alluvial deposits formed by the weathering of these formations. It occurs abundantly in the alluvial clay and sand at the above-named places partly in clearly defined crystals; its hardness is above 6. Sp. gr. 4·35.

Calcite from Sobeslau. In the clay from the subsoil of miocene sand an extraordinary fossil was found resembling petrified wood, but which owed its peculiar structure to a radiated arrangement of the calcite. It much resembles arragonite, but the mean of four determinations of the sp. gr. was 2·67. Its composition is—

CaO.	CO_2.	Fe_2O_3.	H_2O.	Insol.	Total.
53·12	42·63	1·98	0·23	0·19	98·15

Orthoclase from Babitz. The porphyritic granite of the mid-Bohemian granite formation changes after contact with the schists, to a strikingly coarse-grained orthoclase; especially abundant and weathered on the surface at the above-mentioned place. Two varieties can be distinguished by their colour and durability:—a red variety which weathers slowly, and a whitish variety which undergoes change much more readily. An analysis of the latter variety gave—

SiO_2.	Al_2O_3 with Fe_2O_3.	CaO.	K_2O.	Na_2O.	Loss on heating.
63·86	19·42	0·48	12·89	2·06	0·68

Tourmalin from Straschin, Mnichowitz, Sobeslau, Kuhrau. It is widely diffused throughout the primitive rocks. Nevertheless, it is more abundant and more constant in its occurrence where the granite is in contact with the schist. The black crystals from Straschin, measuring as much as 6 cm. long, are often broken off short and cemented by felspar. The tourmalin of Kuhrau occurs in crystals as much as 10 cm. long and 2 cm. thick, often broken and in combination with red orthoclase and potash mica. J. P. L.

Halotrichite from the Tyrol. By J. Hockauf (*Jahrb. f. Min.*, 1888, ii, Ref., 58, from *Verh. geol. Reichsanst.*, 1887, 152—154).—The author has analysed the crusts of a white, yellow, or red colour, which occur as efflorescence on the pyrites-bearing micaceous clay-slate of the Vilnösthal. The specific gravity of the material was 1·98. Analysis gave the following results:—

SO_3.	Al_2O_3.	Fe_2O.	FeO.	MnO.	CaO.	MgO.	K_2O.	H_2O.	Total.
32·97	15·05	6·66	1·77	0·62	0·47	0·10	0·38	41·98	100·00

Of the proportion of water, 27—29 per cent. was given off at 100°, and the remainder at 320—350°. The mineral closely resembles the halotrichite of Idria. B. H. B.

Mineralogical Notes. By A. Frenzel (*Chem. Centr.*, 1888, 492, from *Tschermak's Mitth.*, **9**, 397—400).—In a sample of copiapite, from Valparaiso, a new iron sulphate was found, to which the name *Hohmannite* has been given. It consists of broad-leaved, radial-stemmed aggregates, of a lustrous, chestnut-brown colour. It contains (1)—

	Fe_2O_3.	SO_3.	H_2O.
(1.)	40·05	33·88	29·63
(2.)	37·26	35·58	27·62

and corresponds with the formula $2Fe_2O_3,3SO_3 + 13H_2O$; hardness = 3; sp. gr. = 2·24; streak, yellow ochre colour. When exposed to the air it soon changes into a soft, yellow-ochre-coloured mass, which is insoluble in water, but soluble in weak hydrochloric acid. When dried over calcium chloride, it loses part of its water.

In the same copiapite a second iron sulphate was found, to which the author has given the name *amarantite*. It occurs in very minute

crystals, of a yellowish-red colour. Its composition is given in analysis (2) above; it corresponds with the formula $Fe_2O_3,2SO_3 + 7H_2O$; citron-yellow streak. Sp. gr. 2·11.

Minerals occurring in Ehrenfriedersdorf:—Achroite, colourless tourmalin of the usual form, anatase new to Ehrenfriedersdorf, anglesite, apatite, arsenical pyrites, baryta, chlorite, fluorite, scheelite, wolframite, and tin ore. J. P. L.

New Analysis of Hohmannite. By A. Frenzel (*Chem. Centr.*, 1888, 493, from *Tschermak's Mitth.*, **9**, 401—403).—This analysis shows that hohmannite and amarantite have the same composition—

SO_3.	Fe_2O_3.	H_2O.
33·84	35·58	30·08

and, as Wülfing has shown, almost the same crystallographic and optical properties. Their difference on heating, nevertheless, provisionally prevents their being regarded as identical. J. P. L.

Analyses of Zeolites. By C. Hersh (*Jahrb. f. Min.*, 1888, ii, Ref., 1—5, from *Inaug. Diss.*, Zürich, 1887).—The author has made a series of careful analyses of zeolites. The results are as follows :—

	1.	2.	3.	4.	5.	6.	7.
SiO_2	46·12	46·12	47·36	49·28	43·84	45·72	53·58
Al_2O_3	26·25	28·22	20·13	18·52	20·99	16·79	24·07
CaO.....	14·37	—	8·09	9·36	5·89	—	0·85
BaO	—	—	—	trace	—	22·34	—
Na_2O.....	—	15·87	1·99	0·72	5·78	—	13·60
K_2O	—	—	—	—	1·83	—	—
H_2O	13·89	9·91	22·54	22·02	21·97	15·18	8·29
Total	100·63	100·12	100·11	99·90	100·30	100·03*	100·39
Sp. gr.....	2·25	2·28	2·06	2·08	—	2·35	2·31

	8.	9.	10.	11.	12.	13.
SiO_2.....	58·18	56·91	41·56	39·87	52·24	43·23
Al_2O_3	16·35	15·59	28·23	29·40	25·03	23·41
CaO.....	7·21	7·47	11·39	8·15	—	27·41
Fe_2O_3.....	—	—	—	—	—	1·68
Na_2O.....	2·07	1·14	4·20	8·26	—	—
K.....	—	—	—	—	4·05	—
F.....	—	—	—	—	2·21	—
H_2O.....	16·34	18·73	14·98	14·52	16·61	4·42
Total.....	100·15	99·84	100·36	100·20	100·14	100·15
Sp. gr.....	2·21	2·15	2·25	2·19	2·36	2·91

* 99·93 in original.

1. Scolezite, from Bulandstindr, in Iceland. 2. Natrolite, from Jakuben, in Bohemia. 3. Chabazite, from the Faröe Islands. In dry air this mineral lost 4·87 per cent. of water, and remained constant for a week; in moist air it took it up again. 4. Chabazite, from Oberstein, in the Nahethal. 5. Phacolite, from Richmond, in Victoria. 6. Harmotome, from St. Andreasberg, in the Harz. 7. Analcime, from the Cyclop Islands, near Catania, Sicily. 8. Stilbite, from Djupivogur, in Iceland. 9. Desmine, from Helgustadir, in Iceland. 10. Thomsonite, from Nalsöe, Faröe Islands. 11. Mesolite, from Hauenstein, in Bohemia. 12. Apophyllite, from Bergenhill, in New Jersey. 13. Prehnite, from Harzburg, in the Harz. B. H. B.

Proportion of Carbon and Hydrogen in Carbonaceous Schists: Contribution to the Study of the Formation of Coal. By W. Spring (*Bull. Soc. Chim.*, **49**, 11—16).—From analyses of carbonaceous schist taken from the region of the roof and wall of a bed of coal, at St. Gilles, Liège, it was found that the quantity of silica, alumina, and ferric oxide was practically the same in all samples, but the quantity of carbon and hydrogen varied very considerably. Samples from the roof contained about seven times as much carbon as those from the wall, and in both cases the quantity of carbon diminished as the distance from the bed increased. The quantity of hydrogen found by combustion was very high, owing to the fact that the substance does not give up the whole of its water at 120°; when, however, the water is entirely eliminated, the proportion of carbon to hydrogen was found to be the same in samples from the wall as in the coal itself, from which the conclusion may be drawn that the wall contains particles of coal only slightly altered. In samples from the roof, the proportion of carbon to hydrogen increases as the distance from the coal-bed increases. F. S. K.

Analysis of Lockport Sandstone. By H. W. Weld (*Amer. Chem. J.*, **10**, 224).—The samples of "Medina" sandstone, from Lockport, N.Y., U.S., is characterised by the very high percentage of silica and the small amount of cementing material. It contains—

SiO_2.	Fe_2O_3.	Al_2O_3.	CaO.	
96·41	0·70	1·65	1·02	H. B.

Nepheline-syenite from the Transvaal. By E. A. Wülfing (*Jahrb. f. Min.*, 1888, ii, Mem., 16—34).—This rock occurs at the Zwart-Koppies, in the central Transvaal. It is of a light colour, and has a distinct porphyritic structure. Its primary constituents are orthoclase, plagioclase, augite, nepheline, sodalite, and, in smaller quantities, hornblende, mica, titanite, and opaque ores. The secondary products are hydrated ferric oxide and zeolites. The rock is thus a typical sodalite-bearing nepheline-syenite of porphyritic structure. Analysis gave the following results:—

	TiO_2.	SiO_2.	Al_2O_3.	Fe_2O_3.	FeO.	MnO.	CaO.	MgO.
I.	0·00	43·69	28·64	1·40	—	0·20	2·88	trace
II.	0·26	60·57	13·00	10·15		0·44	2·57	0·60
III.	0·09	53·73	20·35	3·74	2·13	0·51	2·72	0·47

	K_2O.	Na_2O.	H_2O.	Cl.	Total.
I.	3·88	15·17	4·25	0·59	100·70
II.	7·45	3·31	0·59	—	98·94
III.	6·05	7·94	2·02	0·23	99·98

I. Analysis of the portion (39·03 per cent.) soluble in hydrochloric acid. II. Analysis of the insoluble portion. III. Complete analysis. The soluble portion appears to be a mixture of 30 per cent. of zeolite, 59 per cent. of nepheline, and 11 per cent. of sodalite. The Transvaal nepheline-syenite belongs to the group of rocks in which pyroxene is the predominating basic constituent. It is of special interest from the fact that it contains two augites, physically and chemically different, and that both contain alkalis.

B. H. B.

Petrography of the South-western Frontier between Minas and S. Paolo. By J. Machado (*Chem. Centr.* 1888, 496—497, from *Tschermak's Mitth.*, **9**, 313, 360).—The rocks described from the abovementioned district belong to gneiss, different nepheline-syenites, a quartz-augite-diorite, and olivine diabase. The most important are the two first-named groups of rocks, and chief of all the gneisses, because they, by their whole habit, prove themselves to be the real members of the ancient formation. They consist of orthoclase, microcline, microperthite, plagioclase, biotite, quartz, more or less rich hornblende, and the accessory microscopic components, zircon, apatite, and magnetite, and they approximate in their structure sometimes to granite gneisses, and sometimes to augite gneisses. The nepheline-syenites which have been described from other parts of Brazil by Lasaulx and Rosenbusch, compose, according to previous researches, the Plateau of Pocos de Caldas. They are defined outwardly from the adjoining gneiss district by a striking infertility. The partly coarse and partly fine-grained light-grey rocks are composed chiefly of macroscopic orthoclase, nepheline, and augite. The ready decomposition of nepheline gives rise to a generally diffused transformation into a colourless and fibrous substance, with strong double refraction, in which cancrinite is supposed to exist. The green strongly pleochroite augite, which occurs in columns of irregular contour, in radially radiated and fibroglobular aggregates, belongs apparently throughout to the Aegirins.

Titanite occurs frequently; sodalite is locally plentiful, and similar in its habit to ditro; melanite scarce; and lastly, biotite occurs in varying quantities, but only as an accessory. A powdered and broken piece of nepheline-syenite found at Barreiro permitted the isolation of both augite and nepheline. Analyses 1 and 2 respectively:—

	SiO_2.	Al_2O_3.	Fe_2O_3.	FeO.	CaO.	MgO.	K_2O.	Na_2O.	H_2O.
(1.)	51·60	1·92	26·29	4·20	4·25	1·15	1·05	8·89	0·56
(2.)	48·63	25·11	2·58	—	3·52	1·15	1·47	7·50	11·66

The occurrence of modifications which sharply define themselves by a finer grained structure than usual, but which do not differ with regard to their mineral constituents, is noteworthy. The chemical composition of the latter is given in analysis (3). The fine-grained nepheline-syenites appear also, forming massive veins and rocks, similar in outward appearance to compact diorite, and even phonolites, and often coloured completely black, on account of their richness in augite. Wollastonite is sometimes met with in irregular masses, which appear to be as it were eaten away, or in sharply defined crystals, the presence of which was proved optically and chemically by the composition of the hydrochloric acid extract, which is given under analysis (4).

Orthoclase is the chief constituent of these fine-grained syenites, occurring for the most part in opaque twin crystals, according to the Carlsbad law. Next in importance comes nepheline, the amount of which (49·84 per cent.) is shown by the hydrochloric acid extract, and the composition of which is given under analysis (5). Analysis (6) shows the composition of the insoluble residue, which consists chiefly of potash felspar. Analysis (7) is the complete analysis of another fine-grained variety of syenite, in which lavenite was supposed to exist.

Finally, these interesting rocks are represented by perfectly compact and porphyritic gradations, which resemble phonolites much more than the fine-grained varieties, on account of their flat, conchoidal fracture, and their tendency to foliation. They are distinguished on the one hand by the presence of a vitreous base, and on the other hand by a frequently beautiful dendritic, or feather-formed aggregation of their augite.

	SiO_2.	TiO_2.	Al_2O_3.	Fe_2O_3.	MgO.	CaO.	Na_2O.	K_2O.	H_2O.	Totals.
3	52·75	—	22·65	3·65	0·15	1·85	8·10	7·05	3·60	99·70
4	48·21	—	28·89	1·86	0·46	4·11	16·42		0·05	100·00
5	47·31	—	24·19	3·40	0·22	2·80	13·75	5·48	2·86	100·01
6	56·86	—	15·55	8·47	0·28	1·90	1·93	14·63	—	99·62
7	53·10	0·81	22·50	5·10	0·15	2·15	8·49	6·48	1·65	100·43
8	27·73	—	14·50	28·11	8·64	16·85	3·84	3·33	1·01	100·01
9	65·20	—	16·25	5·45	1·87	7·55	2·87	0·50	0·65	100·34

The quartz-augite-diorite forms a massive and extensive mountain range. It is a blackish-green, medium-grained rock, composed in the first line of plagioclase and quartz, then of light-green, partly rhombic, partly monoclinic augite, some hornblende and biotite, and, curiously enough, scapolite, in the form of local deposits, wholly subordinate to these are apatite, magnetite, and pyrites. The composition of the

hydrochloric acid extract proving the admixture of scapolite is shown in analysis (8); the complete analysis in (9). J. P. L.

Hot Springs at Wiesbaden. By R. Fresenius (*J. pr. Chem.* [2], **37**, 465—468).—The author gives a detailed analysis of the water from one of these springs (Kleine Schützenhoff). Its temperature was 45·2° on August 20, 1886, the air temperature being 24·8°. A comparison of the composition of this water with that of two of the other springs, analysed in 1879 and 1885 respectively, shows but slight variations in the constituents. A. G. B.

Organic Chemistry.

Formation of Petroleum. By C. Engler (*Ber.*, **21**, 1816—1827). The theory that petroleum originates from the decomposition of animal matter under high preasure, and at a comparatively low temperature is supported by the author's experiments.

When train-oil is distilled under about 10 atmospheres pressure, which, however, during the operation sank to 4 atmospheres pressure, at an initial temperature of 320°, and a final temperature of slightly above 400°, combustible gases are evolved, and the distillate separates into a lower aqueous, and an upper oily layer. The latter is collected in fractions, and those which are found to contain considerable quantities of unsaturated fatty acids are redistilled. About 60 per cent. of an oily distillate is thus obtained; it has a brownish colour, and in thin layers is transparent with a strong green fluorescence, and a not disagreeable smell. When this oil, which contains more than 90 per cent. of hydrocarbons, is fractioned, and the portion boiling below 150° treated consecutively with concentrated sulphuric acid and a mixture of concentrated sulphuric acid and fuming sulphuric acid, it loses about 37 per cent. by volume. Primary pentane, primary hexane, and primary heptane, were isolated from the residual oil. Primary octane and primary nonane were also present, and probably the mixture also contained secondary hydrocarbons.

When triolein is distilled at about 350° under pressure it yields water, and a mobile oily product, which evolves gas at the ordinary temperature, begins to boil at about 30°, and contains approximately 50 per cent. of compounds boiling below 300°. The residual triolein yields the same products when it is again distilled, so that by repeating the process several times it can be almost wholly converted into volatile and gaseous products.

Tristearin, similarly treated, gives about 60 per cent. of oil, boiling below 300°. Oleic acid and stearic acid, when repeatedly distilled at 350°, under pressure, yield products quite similar to those obtained from triolein and from train-oil. When stearic acid is heated at 350° for several hours in a bent sealed tube, the whole of which is

placed in a digestor, it remains unchanged, only when one limb of the tube, being outside the digestor, allows condensation to take place, is the acid decomposed. On the other hand, oleïc acid and train-oil are decomposed even when the whole of the tube is equally heated.

When train-oil is distilled under diminished pressure, four-fifths of its weight passes over, and the distillate solidifies to a bright yellow, buttery mass, which only contains about 10 per cent. of hydrocarbons. Combustible gases are evolved during distillation, but only a very small quantity of water is formed. F. S. K.

Bromination of Heptane. By F. P. Venable (*Amer. Chem. J.*, **10**, 237—239).—The statement of Schorlemmer that by the action of bromine on normal paraffins only secondary bromides are obtained, is examined, and it is shown that by treating hot heptane with liquid bromine, the secondary bromide is the principal product; that gaseous bromine produces almost equal quantities of the primary and secondary bromides, and that when bromine is dissolved in heptane and allowed to act in the cold, dibromheptanes are the principal products. Both here and in the other methods of bromination, the secondary bromide was never formed alone. H. B.

Action of Chlorous Anhydride on Heptylene. By R. G. Grissom (*Amer. Chem. J.*, **10**, 225—229).—The absorption of the gas is very energetic, and the temperature must be kept low. A small quantity of the product dissolves in water, and this proves to be due to the presence of acetic acid, a solid crystalline acid containing chlorine, and also probably of other fatty acids. The oil insoluble in water was fractionated, but its examination is incomplete; it appears to contain the chlorhydrin $C_7H_{13}ClO$. The heptylene was obtained from heptane from *Pinus sabiniana*. H. B.

Conversion of Œnanthylidene and Caprylidene into Isomeric Hydrocarbons by Means of Alcoholic Potash. By A. Béhal (*Bull. Soc. Chim.*, **49**, 581—584).—*Methylbutylacetylene*, $C_4H_9{\cdot}C{:}CMe$, is formed when œnanthylidene is heated at 140—150° with alcoholic potash for 24 hours. It boils at 112—113° (750·4 mm. pressure), has the specific gravity 0·7632, and does not combine with cuprous chloride nor with an alcoholic solution of silver nitrate. It combines, however, with mercuric chloride, and yields a methylacetone when treated with sulphuric acid.

When chlorœnanthylene is heated at 140—150° with alcoholic potash for 66 hours, a small quantity of œnanthylidene still remains unchanged, but when chlorocaprylene is treated similarly, no caprylidene is present in the product.

When caprylidene is boiled with alcoholic potash for 36 hours, it is completely converted into a hydrocarbon, which is identical with that obtained from caprylene, and, like the latter, does not precipitate cuprous or silver salts, but combines with mercuric chloride and yields a methylacetone when hydrated.

The caprylidene prepared from caprylene (compare *Bull.*, **48**, 704) is probably a hydrocarbon of the true acetylene series. F. S. K.

Alcoholic Silver Nitrate as Reagent for Hydrocarbons of the Acetylene Series. By A. Béhal (*Bull. Soc. Chim.*, **49**, 335—337).—Large quantities of hydrocarbons of the acetylene series contained in a mixture are often undetected by an ammoniacal solution of cuprous chloride or silver nitrate. A 95°, saturated, alcoholic solution of silver nitrate is, however, a most sensitive reagent, but an excess of the solution must be avoided, as the precipitate is sometimes soluble in presence of a large quantity of the reagent. The compounds obtained are mostly crystalline, and seem to be less alterable than those produced with an aqueous ammoniacal solution, from which they also differ in constitution. They are very sparingly soluble in ammonia, especially when they have been prepared for some time, but as a rule no precipitate is formed with an ammoniacal alcoholic solution of silver nitrate.

The precipitate obtained with œnanthylidene contains nitric acid, and has the composition $C_7H_{11}Ag_2NO_3$; it crystallises in acicular prisms, deflagrates when heated, but does not explode by concussion. It blackens when dried at 100° in the air.

The precipitate obtained with acetylene also contains nitric acid, but does not seem to be a definite compound. It is a yellow substance, which explodes by concussion. Substituted hydrocarbons of the acetylene series gave negative results. F. S. K.

Preparation of Isopropylacetylene from Methyl Isopropyl Carbonyl. By A. Béhal (*Bull. Soc. Chim.*, **49**, 23—27).—*Isopropylacetylene*, $Me_2CH{\cdot}C{:}CH$, is prepared by treating methyl isopropyl ketone (dimethylacetone) with phosphorus pentachloride, heating the mixture of chlorides obtained at 130—140° for about six hours with about 10 times its volume of alcoholic potash, and fractioning the product. It is a liquid, and seems to boil at about 30°. It combines instantly with an ammoniacal solution of silver nitrate, forming a white compound, C_5H_7Ag, which crystallises from boiling alcohol in prismatic needles. F. S. K.

Methyliodoform. By P. de Boissieu (*Bull. Soc. Chim.*, **49**, 16—17).—*Methyliodoform*, $CMeI_3$, is prepared by adding the calculated quantity of methylchloroform drop by drop to a carbon bisulphide solution of aluminium iodide; the yield does not exceed 25 to 30 per cent. It crystallises from alcohol in small, yellow octahedra, which melt at 95° with decomposition, is readily soluble in carbon bisulphide, benzene, and ether, rather more sparingly soluble in light petroleum, and sparingly soluble in cold ether. It smells like weak iodoform, and partially decomposes when crystallised from solvents other than alcohol. F. S. K.

Decomposition of Potassium Cyanide. By I. H. Manning (*Amer. Chem. J.*, **10**, 235—236).—The conclusion of v. d. Pfordten that the decomposition and discoloration of strong potassium cyanide

solutions is to be attributed to the action of carbonic acid is not tenable, as shown by Wilkes and confirmed by the author.

H. B.

Potassium Platinocyanide. By T. Wilm (*Ber.*, **21**, 1434—1454).—When a very small quantity of pure nitric acid (sp. gr. about 40° Beaumé) is added to a few drops of a concentrated solution of potassium platinocyanide, the mixture becomes yellowish and, as long as no excess of nitric acid is present, bright yellowish-brown, slender needles, with a copper-coloured lustre, crystallise from the solution; when more nitric acid is added, the crystals redissolve, the solution becomes colourless, and oily drops separate, often without either warming or concentrating the solution. When the whole is evaporated several times with water and exposed to the air, it crystallises to a mass of needles, but no pure product is obtained. When pure nitric acid is added drop by drop to a concentrated solution of potassium platinocyanide, the mixture becomes yellow; after keeping for some time, copper-coloured, lustrous needles separate. This product is washed with cold water, then with alcohol, and dried in the air; it was found to be the chloride, $3[K_2Pt(CN)_4 + 3H_2O],Cl$, the chlorine originating from some impurity in the potassium platinocyanide. The compound $3[K_2Pt(CN)_4],KCN,Pt(CN)_3 + 6H_2O$ is obtained in the form of dark-yellow crystals when pure potassium platinocyanide is treated with nitric acid, as described above (compare Abstr., 1886, 605).

Hydrogen peroxide has no action on a concentrated solution of potassium platinocyanide even when the mixture is warmed, but if a little hydrochloric acid is added to the mixture, the chloride (see above) separates after a short time in bright, copper-coloured crystals. If the solution is evaporated after acidifying with hydrochloric acid and adding more hydrogen peroxide, or if the mother-liquor from the chloride is treated similarly, not a trace of the copper-coloured salt separates, but a colourless, or yellowish, crystalline mass is obtained, which contains the perchloride $K_2Pt(CN)_4Cl_2$.

The bromide is obtained in dark, lustrous needles and prisms when potassium platinocyanide is treated similarly with hydrobromic acid and hydrogen peroxide, but when the mixture is evaporated with an excess of hydrogen peroxide, the perbromide alone is obtained. This is the best method of preparing the bromide, but an excess of hydrobromic acid must be avoided. The pure bromide forms darker needles than those of the chloride; the former crumbles partially on exposure to the air, and when dried becomes white and opaque, but the chloride is stable in the air, and turns a darker greenish-brown when dried.

When pure concentrated sulphuric acid is added drop by drop to a well-cooled, concentrated solution of potassium platinocyanide, hydrogen potassium sulphate separates and the liquid becomes yellow. When the solution is filtered and the residue washed with alcohol, orange-red needles with a violet tint crystallise at the surface of contact of the yellow filtrate and the alcohol, but on adding more alcohol, the crystals are redissolved. Yellowish-red needles with a deep violet to steel-grey tint can be observed when a portion of the mixture is evaporated and examined under the microscope on a warm

glass, but they soon become pale yellow and deliquesce to an oily liquid, which when treated with absolute alcohol crystallises to a mass of orange-red needles. On account of the extreme solubility of this compound in water, alcohol, and ether, it could not be obtained pure.

The action of a mixture of sulphuric acid and hydrogen peroxide on potassium platinocyanide seems to be influenced by the temperature and by the concentration or quantity of the reagents. When sulphuric acid is gradually added to a well-cooled mixture of a concentrated solution of potassium platinocyanide and hydrogen peroxide, slender, dark brown, lustrous needles separate after a few hours' time. This product was washed with water, then with alcohol, and dried in the air, but its composition could not be ascertained. A second experiment, in which pure potassium platinocyanide was employed, yielded the same dark brownish-yellow needles. The product was obtained in the pure state, and was found to be identical with the compound previously obtained by acting on pure potassium platinocyanide with nitric acid.

This same compound is obtained when a cold, concentrated solution of pure potassium platinocyanide is electrolysed; hydrogen is evolved at the negative pole, and a mass of yellowish-brown crystals separates at the positive pole, but no evolution of oxygen is evident. The crystals are washed with water, then with alcohol, and dried in the air.

The constitution of this compound is $3[K_2Pt(CN)_4],KCN,Pt(CN)_3 + 6H_2O$; it liberates iodine from potassium iodide.

The chloride and the bromide referred to above have an analogous constitution, namely, $2[K_2Pt(CN)_4],2KCN,Pt(CN)_2Cl + 9H_2O$ and $2[K_2Pt(CN)_4],2KCN,Pt(CN)_2Br + 9H_2O$ respectively.

F. S. K.

Metallic Nitroprussides. By T. H. Norton (*Amer. Chem. J.*, **10**, 222—224).—The following salts are described: $CdFe(CN)_5NO$, $Hg_2Fe(CN)_5NO$, $CoFe(CN)_5NO + 4H_2O$, and $NiFe(CN)_5NO$. All are insoluble in water and, with the exception of the cadmium salt, decompose easily; the cobalt salt is thrown down as a crystalline powder, the other three are amorphous. H. B.

Combination of Aluminium Chloride with Acetonitrile, Chloracetonitrile, and Trichloracetonitrile. By P. Genvresse (*Bull. Soc. Chim.*, **49**, 341—343).—The compound $CNMe,Al_2Cl_6$ separates as a white, crystalline powder, when aluminium chloride is added gradually to acetonitrile kept cold by water at the ordinary temperature. When heated, it partially decomposes, becomes pasty, and melts to a brown liquid which solidifies to a resinous product on cooling. On continued heating, or when treated with water, it is completely decomposed. The compound $CN\cdot CH_2Cl.Al_2Cl_6$ is obtained by adding chloracetonitrile gradually to aluminium chloride and warming the mixture. The product melts to a greyish-brown liquid which crystallises on cooling. It is very readily soluble in benzene, melts about 38°, and decomposes when heated or when treated with water.

The compound $CN{\cdot}CCl_3,Al_2Cl_6$ is formed when aluminium chloride is added gradually to trichloracetonitrile. It is a white, crystalline substance, which quickly decomposes when heated, and has no well-defined melting point. It is sparingly soluble in benzene, and is decomposed when treated with water. F. S. K.

Compound of Methyl Alcohol and Potash. By C. Göttig (*Ber.*, **21**, 1832—1835).—The compound $5CH_4O,3KOH$, obtained when a methyl alcoholic solution of potash is evaporated, crystallises in transparent, elastic plates melting at about 110°, which, when placed on water, move about rapidly on the surface and are decomposed. When kept in a desiccator over sulphuric acid, it gradually gives up the greater part of its alcohol and becomes opaque, but does not entirely lose its property of moving about on the surface of water. F. S. K.

Derivatives of Isodulcitol. By W. Will and C. Peters (*Ber.*, **21**, 1813—1815).—A *lactone*, $C_6H_{10}O_5$, is produced when isodulcitol is treated with bromine in aqueous solution. It crystallises in needles, melts at 148°, and is readily soluble in alcohol and water, but sparingly soluble in ether. It behaves similarly to saccharin, but differs from the latter in melting point, solubility, rotatory power, and crystalline form. An oily mixture of a lactone, boiling at 206—212°, and a liquid, fatty acid is obtained when it is boiled with concentrated hydriodic acid and a little phosphorus. It yields amorphous *calcium isodulcitonate*, $(C_6H_{11}O_6)_2Ca$, when boiled with calcium carbonate; the *barium* salt, prepared similarly, is also amorphous.

The authors also prepared isodulcitolcarboxylic acid, its barium and calcium salt, and the corresponding lactone melting at 169°. (Compare Fischer and Tafel, this vol., p. 806). F. S. K.

Rotatory and Reducing Power of Lactose. By G. Denigès and E. Bonnans (*J. Pharm.* [5], **17**, 363—367, 411—414).—The figures given by different authors for the rotatory power of lactose vary considerably. In a recent work, Schmöger gives results obtained with solutions containing 4 to 36 per cent. at a temperature t for lactose hydrate as $\alpha = 52°{\cdot}53 + (20 - t) \times 0{\cdot}055$, which for anhydrous lactose is $\alpha = 55°{\cdot}30$ at 20°. The results obtained by the authors after three or more crystallisations of the lactose agree perfectly with Schmöger's results. In both cases the penumbra polarimeter with monochromatic light was employed. The rotation is strictly proportional to the dilution of the solution, and the correction for temperature is also dependent on the dilution; thus the correction for a deviation ρ at temperature t, to get the deviation at 20° is

$$x = \rho \frac{\rho}{1000} \times (20 - t) = \rho \times \frac{1020 - t}{1000}.$$

The results obtained with Soleil's saccharimeter are 4° to 6° higher than those given above.

There is considerable diversity in the published results as to the reducing power of lactose. 10 c.c. of Fehling's solution diluted to

20 c.c. and reduced by 0·049 gram of glucose is equally reduced by 70·2 c.c. of hydrated lactose solution of 1 gram per litre. Hence 5 grams glucose is equivalent to 7·16 grams hydrated lactose, or to 6·8 grams anhydrous lactose. Fehling's solution prepared from different formulæ gives the same result. J. T.

Mannose. By E. Fischer and J. Hirschberger (*Ber.*, **21**, 1805—1809).—Mannosephenylhydrazone, $C_{12}H_{18}N_2O_5$ (Abstr., 1887, 567), is prepared by heating 200 grams of mannitol dissolved in 1300 c.c. of water with 650 c.c. of nitric acid (sp. gr. = 1·4) for eight hours at 42°. After one or two days, soda is added to neutralise the greater portion of the nitric acid, whilst the nitrous acid is got rid of by adding about 30 grams of urea. The mixture is then completely neutralised with soda and treated with a solution of 100 grams of phenylhydrazine in very dilute acetic acid. After half an hour, it is filtered from the crystalline precipitate, which is washed with water, pressed, and then washed with acetone, after which it is colourless. It crystallises from water in very slightly yellowish, slender prisms soluble in 80 to 100 parts of hot water, much less soluble in alcohol and acetone. It melts between 195° and 200° (not 188°). The dilute hydrochloric acid solution is lævorotatory.

Mannose, $OH{\cdot}CH_2{\cdot}CH(OH){\cdot}CO{\cdot}CH(OH){\cdot}CH(OH){\cdot}CH_2{\cdot}OH$, is obtained by dissolving the above phenylhydrazone in four parts of hydrochloric acid (sp. gr. = 1·19) cooled with ice and salt. After a quarter of an hour, the product is filtered from the phenylhydrazine hydrochloride which separates, treated with twice the volume of ice-water, and neutralised with lead carbonate. The filtrate, which contains, besides mannose, lead chloride and phenylhydrazine, is freed from the latter substances and evaporated in a vacuum. It is then dissolved in absolute alcohol and precipitated with absolute ether. It forms a light yellow syrup having all the properties of the glucoses; it has a sweet taste, dissolves readily in water and alcohol, reduces Fehling's solution, and yields humic substances when heated with 20 per cent. hydrochloric acid. The aqueous solution is dextrorotatory, but much less so than dextrose. When a 10 per cent. solution of mannose is treated with 2 per cent. sodium amalgam, mannitol is formed.

Phenylmannosazone, $C_{18}H_{22}N_4O_4$, is formed when mannosephenylhydrazone (1 part), phenylhydrazine hydrochloride (3 parts), sodium acetate (4 parts), and water (80 parts), are heated at 100—105° for 4 to 5 hours. It crystallises from alcohol in slender, yellow needles, generally grouped in globular aggregates, and melts at 210°.

N. H. M.

Glycogen in Lower Animals: Compounds of Glycogen with Sulphuric Acid. By F. Anderlini (*Chem. Centr.*, 1888, 451—452, from *Atti. R. Inst. Veneto* [6], **5**, 1291—1294).—The author has been able to extract glycogen from *Bombyx mori* in the different periods of its life. The greatest amount was found in the chrysalis; a little less in the female butterfly. A small quantity of glycogen was also found in *Blatta orientalis*.

Concentrated sulphuric acid dissolves glycogen in the cold without

perceptible coloration. When, after prolonged standing, the solution was diluted with water, neutralised with barium carbonate, and concentrated in a vacuum, amorphous residues were obtained, the aqueous solution of which became turbid on heating. Glycogen dissolves also in chlorosulphonic acid; the solution of 1 part in 6 to 7 parts chlorosulphonic acid, becomes sticky after 24 to 28 hours, later solid and crystalline. This substance, when dried on porous plates, forms colourless, very hygroscopic crystals, which readily decompose when exposed to the air. The amorphous lead and barium salts are obtained by evaporating the solution which has been neutralised with lead and barium carbonates respectively. J. W. L.

Succinenylamidoxime and some of its Derivatives. By F. Sembritzki (*Chem. Centr.*, 1888, 623—624).—*Succinenylamidoxime*, $C_2H_4[C(NH_2):N\cdot OH]_2$, is obtained by mixing equivalent quantities of hydroxylamine hydrochloride and sodium carbonate with an alcoholic solution of cyanobenzoic acid; it melts with blackening and disengagement of ammonia at 188°, is soluble in hot water, only slightly soluble in alcohol, and insoluble in chloroform, benzene, and light petroleum. It forms both acid and basic salts. The acetyl-derivative melts at 167—168°. The benzoyl-derivative melts at 192°, and when heated with water in a sealed tube at 150° to 160° for five hours, furnishes *succinenylazoxybenzene*, $C_2H_4C_2\left(\begin{matrix}NO\\ N\end{matrix}\!\!>CPh\right)_2$, melting at 158° to 159°.

Succinenylamidoxime ethyl ether, $C_2H_4[C(NH_2):NOEt]_2$, results from the reaction of succinenylamidoxime, ethyl iodide, and sodium ethoxide, and melts at 119°.

Succinenyluramidoxime, $C_2H_4[C(N\cdot OH):NH\cdot CO\cdot NH_2]_2$, was prepared from equivalent proportions of succinenylamidoxime and potassium cyanate in concentrated watery solution. It contains water of crystallisation, which is given off at 100° to 105°; when anhydrous it melts with browning and decomposition at 163·5°.

If an alcoholic solution of cyanosuccinic acid is digested with hydroxylamine and sodium carbonate, and then evaporated to the point of crystallisation on a water-bath, *succinenylimidoxime*, $C_2H_4<\begin{matrix}C(NOH)\\ C(NOH)\end{matrix}>NH$, separates out. This substance, dissolved in soda, gives first a blue and then a green colour; with ferric chloride it gives a dark violet coloration. The *benzoyl*-derivative melts at 187—189°, the *acetyl*-derivative at 170—171°. J. P. L.

Acetals of Normal Propyl Glycol. By H. Lochert (*Bull. Soc. Chim.*, **49**, 352—354).—The compound, $CH_2<\begin{matrix}CH_2\cdot O\\ CH_2\cdot O\end{matrix}>CHMe$, is obtained by heating a mixture of acetaldehyde (1 mol.) with propyl glycol (2 mols.) and fractioning the product. It is a colourless liquid which boils at 110—112° (768·6 mm. pressure), and has a slight smell of acetaldehyde; it dissolves in $1\frac{1}{2}$ times its volume of water, and is very readily soluble in alcohol and ether. It is separated from its aqueous solution by calcium chloride. Caustic alkalis do not act on

the dry compound, but it yields acetaldehyde and propyl glycol when heated with water in sealed tubes, or, more quickly, when treated with alkalis or acids.

The compound, $CH_2<\begin{matrix}CH_2\cdot O\\CH_2\cdot O\end{matrix}>CH\cdot C_4H_9$, is obtained similarly from propyl glycol and valeraldehyde. It is a colourless, mobile liquid which boils at 164—166°, and has a faint smell of valeraldehyde. It is almost totally insoluble in water, and resembles the preceding compound in all its properties. F. S. K.

Methylhydrazine. By G. v. Brüning (*Ber.*, **21**, 1809—1810).—*Nitrosomethylcarbamide*, $NH_2\cdot CO\cdot NMe\cdot NO$, prepared by treating a cold, neutral, aqueous solution of methylcarbamide with sodium nitrite, crystallises in yellowish leaves, melts at 123—124° with decomposition, and is readily soluble in alcohol and warm water. It is decomposed by continued boiling with water, and is to a large extent converted into methylhydrazinecarbamide when treated with zinc and acetic acid in alcoholic solution.

Methylhydrazine sulphate, $NHMe\cdot NH_2,H_2SO_4$, is obtained when the alcoholic solution of methylhydrazinecarbamide is evaporated with hydrochloric acid and the residue heated at 100° with concentrated hydrochloric acid. It crystallises from alcohol in shining needles. The free *base* is a colourless liquid with a strongly ammoniacal smell; it shows all the reactions of ethylhydrazine. F. S. K.

Action of Silicon Tetrafluoride on Acetone. By T. H. Norton and J. H. Westenhoff (*Amer. Chem. J.*, **10**, 209—213).—Silicon tetrafluoride was passed into acetone, and the product distilled fractionally; a large proportion of the acetone was unaltered, the fractions boiling higher than acetone and below 90° all decomposed readily after a time, and the only portions that were stable and were analysed were two distilling between 90° and 100°, their specific gravities were 0·904 and 1·36. The analyses present, however, many difficulties as regards the determination of silicon, fluorine, and oxygen; the lighter liquid contains about 11 per cent. of hydrogen, 62·5 per cent. of carbon, and 2 per cent. of silicon, with seemingly no fluorine; whilst the heavier contains 10 per cent. of hydrogen, 10 per cent. of carbon, 5·5 per cent. silicon, and large quantities of fluorine and probably oxygen.

H. B.

Bromination of Acetone: Action of Monobromacetone on Ammonium Thiocyanate. By T. H. Norton and J. H. Westenhoff (*Amer. Chem. J.*, **10**, 213—216).—The additive product of acetone and bromine described by Linnemann could not be obtained, nor could more than 50 per cent. of the acetone be converted into dibromacetone, whereas Mulder has reported that tetrabromacetone is obtainable. Whilst chloracetone and ammonium thiocyanate easily react to yield the thiocyanate of thiocyanisopropimine, bromacetone does not, or at any rate the product of the reaction cannot be isolated from the tarry matters formed. H. B.

Action of some Organic Acids on Ethyl Oxalate. By LORIN (*Bull. Soc. Chim.*, **49**, 344—345).—Oxalic acid, formic acid, and acetic acid act on normal ethyl oxalate at 140°, and under absolutely similar conditions, forming the ethyl salt of the acid employed and ethyl formate with evolution of carbonic anhydride and small quantities of carbonic oxide. In the case of oxalic acid, the yield of ethyl formate is twice as large as with acetic or formic acid. Benzoic acid and ethyl oxalate do not react even at 240°. F. S. K.

Action of Silver on Ethyl Dibromosuccinate. By J. GORODETZKY and C. HELL (*Ber.*, **21**, 1801—1802).—Ethyl dibromosuccinate dissolved in light petroleum (b. p. 60—70°) is slightly warmed with an equal amount of dry, finely-divided silver (prepared by reducing silver chloride with iron) for two to three hours. The liquid is poured off from the silver bromide and again warmed with silver until no more action takes place. The product of the reaction is pure ethyl fumarate. N. H. M.

Ethyl Cyanosuccinate and Ethyl Cyanotricarballylate. By A. HALLER and L. BARTHE (*Compt. rend.*, **106**, 1413—1416).—A mixture of ethyl cyanacetate, sodium ethoxide, and ethyl monochloracetate, in equivalent proportions, dissolved in excess of alcohol, was boiled until the liquid was no longer alkaline. It was then filtered to remove sodium chloride, heated to expel excess of alcohol, treated with ether, and the product distilled in a vacuum. *Ethyl cyanosuccinate*, $COOEt{\cdot}CH_2{\cdot}CH(CN){\cdot}COOEt$, is a colourless, oily liquid which boils at 157—160° under a pressure of 14 mm., and is insoluble in water, but soluble in alcohol, ether, and alkalis. With sodium ethoxide it yields a derivative, $COOEt{\cdot}CH_2{\cdot}CNa(CN){\cdot}COOEt$.

In the preparation of the preceding compound a product is obtained which boils at 185—200°, and crystallises in the receiver, or may be crystallised by strongly cooling the distillate. This is *ethyl cyanotricarballylate*, $COOEt{\cdot}C(CN)(CH_2{\cdot}COOEt)_2$, which can be purified by recrystallisation from alcohol, and forms hard, white crystals, which melt at 40—41°, and dissolve in alcohol and ether, but are insoluble in water or alkalis. It is probable that in the preparation of the cyanosuccinate some ethyl cyanosodiosuccinate is formed, and this reacts with the chloracetate to form the cyanotricarballylate. The latter can actually be prepared from the cyanosuccinate, sodium ethoxide, and ethyl monochloracetate.

Cyanotricarballylic acid is the mononitrile of isoallylenetetracarboxylic acid. C. H. B.

Influence of Certain so-called Negative Radicles on the Functions of Certain Groups. By A. HALLER (*Bull. Soc. Chim.*, **49**, 31—36; compare V. Meyer, this vol., p. 147, and Haller, Abstr., 1882, 1189).—The author in his investigations on the influence of certain radicles on the functions of certain groups, has prepared ethyl cyanomalonate, ethyl acetylcyanoacetate, ethyl benzoylcyanoacetate, and the propionyl, butyryl, orthotolnyl, and phenylacetyl-derivatives of ethyl cyanoacetate by treating the sodium compound of ethyl

cyanoacetate with acid chlorides. The orthotoluyl- and phenylacetyl-derivatives yield cyanoketones when treated with boiling water.

Ethyl sodiocyanacetate and ethyl chloracetate yield a crystalline compound which is probably ethyl cyanosuccinate. Metallic sodium dissolves in methyl phenylacetate when the mixture is gently warmed.

When the lead salt of phenylaceticorthocarboxylic acid is distilled, carbonic anhydride and a reddish-coloured liquid, boiling above 200°, are produced.

A resinous product is obtained when ethyl orthonitrophenylacetate is treated with sodium ethoxide.

Ethyl succinimidoacetate yields a sodium-derivative which reacts with benzoic chloride.

Ethyl trichlorolactate shows an acid reaction with turnesole, and reduces ammoniacal silver and Fehling's solutions; its acetyl-derivative has none of these properties, and is quite insoluble in alkalis.

F. S. K.

Molecular Refraction of the Isomerides Fumaric and Maleïc Acids; Citraconic, Itaconic, and Mesaconic Acids, and of Thiophen. By C. KNOPS (*Chem. Centr.*, 1888, 464—465, from *Verh. nat. Ver. Bonn*, 1887, 17).—The isomeric compounds which cannot be explained by the theory of valency, such as the physical isomerides and such isomerides as seem to have the same chemical composition yet yield different derivatives when treated with the same reagents (optically active and optically inactive substances), were compared by the author with reference to those physical properties which appeared to be correlated to substances of known constitution.

From his experiments it follows that fumaric and maleïc acids, on the one hand, and mesaconic and citraconic acids on the other hand, probably possess different structures. The author also submits a new formula for thiophen. No details are given. J. P. L.

Action of Normal Alkaline Tungstates on Solutions of Tartaric Acid. By D. GERNEZ (*Compt. rend.*, **106**, 1527—1530; compare Abstr., 1887, 540, and this vol., p. 97).—Crystallised sodium tungstate, Na_2WO_4, was added to 1·25 gram of tartaric acid in quantities varying from one-fortieth to 17 mols. per molecule of acid, the proportion of water being sufficient to make the volume of the liquid up to 50 c.c. The rotatory power of the solution increases with the proportion of tungstate until the salt and the acid are present in equal molecular proportions. It then increases more and more slowly until 2 mols. of the salt are present for each molecule of acid, beyond which point the rotatory power decreases by gradually diminishing quantities. These facts indicate the formation of a compound of 1 mol. tartaric acid and 1 mol. sodium tungstate and a second compound containing 1 mol. of the acid and 2 mols. of the tungstate; the maximum rotatory power being 22 times as great as that of the tartaric acid in the solution. When further quantities of the tungstate are added, the compound of the salt and the acid is gradually dissociated.

With potassium tungstate, the phenomena indicate the formation of a compound of 1 mol. of the tungstate and 1 mol. of tartaric acid,

the maximum rotation being 23 times as great as that of the tartaric acid in the solution. C. H. B.

Action of Chlorine on Sulphonic and Oxythio-derivatives of Heptyl. By W. Spring and C. Winssinger (*Bull. Soc. Chim.*, **49**, 68—74; compare Abstr., 1884, 1127).—*Dichlorheptylsulphonic acid* is formed by acting on normal heptylsulphonic acid with chlorine in direct sunlight for seven to eight hours; not a trace of sulphuric acid is formed during the reaction. The *barium* salt has the formula $(C_7H_{13}Cl_2SO_3)_2Ba$.

Trichlorheptylsulphonic acid is prepared by treating the preceding compound with chlorine until no further action takes place. It is readily decomposed by boiling water with formation of sulphuric acid and a chlorheptyl-derivative.

Hexachlorhydroxyheptylic acid, $C_7H_8Cl_4O_3$, is obtained when heptylsulphonic acid is heated with iodine trichloride at 167—170°; it is insoluble in water.

Pentachlorheptylic acid, $C_7H_9Cl_5O_2$, and trichlorheptylsulphonic acid, identical with the trichloro-derivative described above, are also produced by the same reaction. Both compounds are soluble in water, and yield barium salts which crystallise together to form a substance, $2(C_7H_{12}Cl_3SO_3)_2Ba,3(C_7H_5Cl_3O_2)_2Ba + 24H_2O$.

When heptyloxysulphide is treated with chlorine in presence of water, an oily product is obtained, about two-thirds of which is soluble in soda. Trichloro- and tetrachloro-heptane were isolated from the insoluble residue; both compounds give silver œnanthylate, œnanthaldehyde, a small quantity of heptyl œnanthylate, and a deposit of silver when treated with silver oxide and water at 155—162°. The soda solution yielded a mixture of two barium salts, $2(C_7H_{14}ClSO_3)_2Ba$, $(C_7H_{13}Cl_2SO_3)_2Ba$ and

$$2(C_7H_{14}ClSO_3)_2Ba.(C_7H_{15}SO_3)_2Ba;$$

these were separated by crystallisation. F. S. K.

3 : 4-Thioxen and Tetramethylthiophen. By N. Zelinsky (*Ber.*, **21**, 1835—1839).—*ββ-Thioxen*, $C_4SH_2Me_2$ [$Me_2 = 3 : 4$], is obtained when a mixture of sodium dimethylsuccinate and phosphorus trisulphide is distilled. It is a transparent, strongly refractive oil which boils at 144—146° (762 mm. pressure), and has a characteristic thiophen-like smell. A trace of this compound, dissolved in concentrated sulphuric acid, gives an intensely emerald-green solution with isatin: a glacial acetic acid solution gives a greenish-violet coloration with anthraquinone.

From the product obtained by the oxidation of 3 : 4-thioxen an acid was isolated which crystallises in very slender needles, melts at 139°, and is sparingly soluble in cold water, and sublimes.

Tetramethylthiophen, C_4SMe_4, prepared by converting trimethylthiophen into its iodo-derivative by means of iodine and mercuric oxide and then treating the product with sodium and methyl iodide in ethereal solution, boils at 182—184° (uncorr.). The yield is extremely small. F. S. K.

Constitution of Benzene. By A. Claus (*J. pr. Chem.* [2], **37**, 455—464; compare Abstr., 1887, 719).—In this paper, the author replies to the criticisms passed by Baeyer on his diagonal formula (*Annalen*, **245**, 120), and maintains that Baeyer's work on terephthalic acid and its hydrogen additive derivatives supports this formula. The author proceeds to explain orientation and the structure of quinone by the help of his formula for the benzene nucleus.

A. G. B.

Iodation of the Phenols by Nitrogen Iodide. By C. Willgerodt (*J. pr. Chem.* [2], **37**, 446—448).—By the action of nitrogen iodide on dilute alcoholic potassium phenoxide, ammonia is evolved, and triiodophenol is formed together with a little iodophenol. The author describes the best method of handling the nitrogen iodide in order to avoid an accident. Powdered iodine in dilute alcoholic phenol, mixed with ammonia, produces orthiodophenol, together with tri-, di-, and paramono-iodophenol. If β-naphthol be substituted for phenol, iodo-β-naphthol, previously described by Meldola (Trans., 1885, 525), is obtained by both methods, together with two secondary products, one crystallising in needles melting at 223°, the other an amorphous, greyish-white substance. By applying this method to the iodation of the phenols, the author and Kornblum have obtained *iodothymol*, $C_6H_2MePrI{\cdot}OH$ [1 : 4 : 6 : 3] melting at 69°, and *di-iodorthocresol* melting at 69·5°. A. G. B.

Action of Alkali Nitrites on Halogen-derivatives of Quinones. By F. Kehrmann (*Ber.*, **21**, 1777—1783).—When trichlor- or tribrom-orthotoluquinone is digested on a water-bath with potassium nitrite (5 mols.), a heavy, sandy, crystalline precipitate is formed, which, when dissolved in hot water and treated with barium chloride, yields the barium salt of a *nitrodihydroxytoluquinone*,

$$C_7H_5NO_6\ [O_2 : (OH)_2 : Me : NO_2 = 1 : 4 : 3 : 6 : 2 : 5].$$

This when oxidised with cold nitric acid is converted into what is probably the *methylhydroxypentaketone*, $C_6O_5Me{\cdot}OH$. The latter is dissolved by cold, strong sulphurous acid (similarly to triquinoyl, Nietzki and Benckiser, Abstr., 1885, 781); on neutralising the solution with sodium carbonate, a reddish-yellow liquid is formed containing probably methylrhodizonic acid [O_3 : OH : OH : Me,OH = 1 : 4 : 5 : 3 : 6 : 2].

Dichloro-α-naphthaquinone, when heated with an aqueous alcoholic solution of sodium nitrite, yields the sodium salt of Diehl and Merz's nitronaphthalic acid (hydroxynitro-α-naphthaquinone) (Abstr., 1878, 888), which must therefore have the constitution

$$C_{10}H_4O_2(OH){\cdot}NO_2\ [O_2 : OH : NO_2 = 1 : 4 : 3 : 2].$$

When this is reduced with stannous chloride, *amidotrihydroxynaphthalene*, [$OH_3 : NH_2 = 1 : 3 : 4 : 2$], is formed. When amidotrihydroxynaphthalene hydrochloride is dissolved in cooled, strong nitric acid, and the whole poured into 10 parts of nearly boiling water, the compound $C_{10}H_4O_3{\cdot}N{:}NO$ [$(OH)_3 : N_2O = 1 : 3 : 4 : 2$], separates in splendid, reddish-yellow needles. The latter dissolves in hot water and alcohol,

but is decomposed by prolonged boiling; nitric acid dissolves it very readily. When heated at 100° it detonates.

Dichloro-α-naphthaquinonesulphonic acid (Claus and S. van der Cloet, this vol., p. 602) is converted by alkali nitrite into a nitrohydroxy-α-naphthaquinonesulphonic acid, [O_2 : NO_2 : OH = 1 : 4 : 2 : 3]. The *potassium salt* forms readily soluble, yellow, hair-like needles: the *barium salt* is a yellow, crystalline precipitate, sparingly soluble in cold water. N. H. M.

Formation of Eugenol from Coniferin. By L. Chiozza (*Chem. Centr.*, 1888, 443, from *Rendi. R. Inst. Lombardo* [2], **21**, 172).—Conferin is reduced to eugenol by sodium amalgam in a weak alkaline solution. Some unchanged coniferin is precipitated from the cold solution by adding a little water. Dilute sulphuric acid precipitates the eugenol from the filtered solution. As an intermediate product coniferyl alcohol, $C_{10}H_{12}O_3$, is formed. J. W. L.

Combinations of Aniline with Cuprous Chloride, Bromide, and Iodide. By A. Saglier (*Compt. rend.*, **106**, 1422—1425).—2 grams of cuprous chloride was heated in sealed tubes at 100° with 40 c.c. of a mixture of equal volumes of alcohol and aniline. When cooled the liquid deposits white, acicular crystals of the composition $2PhNH_2,Cu_2Cl_2$, which rapidly turn green when exposed to the air. The bromide under similar conditions yields an analogous compound. The corresponding iodine compound forms white needles which become rose-coloured and finally black when exposed to the air.

These compounds are not affected by light, but are decomposed by alkalis with liberation of aniline. They are insoluble in water and alcohol, and alter rapidly when exposed to air, but if freed from excess of aniline, they can be preserved for some time. At 50° they begin to decompose with evolution of aniline, and at a dull red heat a residue of the cuprous salt is left.

When equivalent quantities of cuprous chloride and aniline hydrochloride are heated in sealed tubes with dilute hydrochloric acid until the copper salt is dissolved, the liquid when cooled deposits white crystals of the compound $2PhNH_3Cl,Cu_2Cl_2$, which is more stable than the preceding compounds, but becomes first rose-coloured, then brown, and finally black when exposed to air. Cuprous bromide yields a similar compound which crystallises in lamellæ if the solution is cooled rapidly, and in prismatic needles if it is cooled slowly.

C. H. B.

Action of Fluoride of Silicon on Organic Bases. By A. M. Comey and C. L. Jackson (*Amer. Chem. J.*, **10**, 165—181).—The action of silicon tetrafluoride on ammonia results in the formation of the compound $2NH_3,SiF_4$ (Gay-Lussac and Thenard); the action on aniline has only been imperfectly studied by Laurent and Delbos and by Knop. It is now shown that additive products of a somewhat different type, namely, $3NH_2X,2SiF_4$, are formed by the following amines:—Aniline, paratoluidine, orthotoluidine, parachloraniline, diphenylamine, dimethylaniline, quinoline, and dimethylamine, and besides these

aniline and dimethylamine from another compound, of the type $4NH_2X,2SiF_4$. The compound $3NH_3,2SiF_4$ could not be prepared.

Trianiline disilicotetrafluoride, $3NH_2Ph,2SiF_4$, is formed very readily, is amorphous, sublimes without melting, is decomposed slowly by alcohol, quickly by water with formation of aniline silicofluoride, which is reconverted into the original substance by sublimation. Ammonia decomposes it with formation of the diammoniosilicotetrafluoride of Gay-Lussac. The corresponding compounds of orthotoluidine, paratoluidine, monochloraniline, and parabromaniline, are very similar in all respects; symmetrical tribromaniline undergoes no reaction, neither do diphenylamine, dimethylaniline, or quinoline until they are dissolved in benzene; in the last cases the compounds are crystalline. The tri-dimethylamine-derivative is only obtained by subliming *di-dimethylamine silicotetrafluoride*. This is deposited as a white powder on bringing together its constituents; it is at once decomposed by heat, yielding dimethylamine and the tri-compound. The aniline salt, $4NH_2Ph,2SiF_4$, is obtained by leading aniline vapour into silicon tetrafluoride, or by heating the trianiline compound with aniline at 150°; it is not very stable, decomposing even at ordinary temperatures into trianiline disilicotetrafluoride, and aniline; for this reason the simplest empirical formula is doubled.

It is possible that the pyridine and quinoline additive products obtained with silicon tetrachloride by Harden (Trans., 1887, 40) correspond with the above, although they are less stable. H. B.

Action of Phenylamine and Diphenylamine Hydrochlorides on some Fatty Amines. By H. Eckenroth and J. Rückel (*Chem. Centr.*, 1888, 784, from *Pharm. Zeit.*, **33**, 282).—Phenylurethane does not result from the action of phenylamine hydrochloride on urethane as would be expected, but diphenylcarbamide, $CO(NHC_6H_5)_2$, is formed. From analogy, diphenylamine hydrochloride and urethane should yield tetraphenylcarbamide, but diphenylamine is the only product; the tetraphenylcarbamide may probably be decomposed when in a nascent state by the hydrochloric acid formed by the reaction. J. P. L.

Double Compounds of Acetone with the Sulphites of Aromatic Amines. By P. Boessneck (*Ber.*, **21**, 1906—1910).—When sulphurous anhydride is passed into acetone, the gas is absorbed with considerable development of heat. The product, probably $CMe_2<^{O}_{O}>SO$, when poured into water, sinks, and only mixes with the water when violently shaken with it; when heated, it decomposes into its constituents.

Acetone-aniline sulphite, $COMe_2,SO_2,NH_2Ph$, is obtained by adding aniline to acetone saturated with sulphurous anhydride, and washing the crystalline product with acetone. If $1\frac{1}{2}$ mol. of alcohol is added to the acetone before treating with aniline, the double compound separates in stellate groups of long, colourless needles. It is very readily soluble in water, soluble in alcohol, and insoluble in water. When kept in closed vessels, it remains for months unchanged.

Acetone-dimethylaniline sulphite, $COMe_2,SO_2,NMe_2Ph$, forms lustrous plates readily soluble in alcohol and water, insoluble in acetone.

Acetone-ethylaniline sulphite, $COMe_2,SO_2,NHEtPh$, forms very large crystals.

Acetone-ethylenetoluylenamidine sulphite, $C_3H_6O,SO_2,C_9H_{10}N_2$, forms lustrous, rhombic crystals.

Aniline sulphite, NH_2Ph,SO_2, can be obtained in well-formed plates by passing sulphurous anhydride through a warm aqueous solution of equal parts of aniline and potash; when removed from the solution, it at once gives off sulphurous anhydride (compare Schiff, *Annalen*, **140**, 133, and **144**, 47). N. H. M.

Derivatives of Paraphenolphenylamine. By E. v. Bandrowski (*Monatsh.*, **9**, 133—137; compare *Monatsh.*, 1887, 523).—When paraphenolphenylamine, $OH\cdot C_6H_4\cdot NHPh$ (1 mol.) is dissolved in benzene and oxidised with yellow mercuric oxide (2 mols.), two atoms of hydrogen are removed with formation of the compound

$$C_6H_4<\begin{matrix}N\cdot Ph\\O\text{——}\end{matrix}>.$$

This substance crystallises in interwoven masses of a bright red colour melting at 97°. It is extremely soluble in alcohol, ether, chloroform, and benzene, is best recrystallised from light petroleum, and readily decomposes on boiling with water, alkalis, or dilute acids with formation of quinone. In a similar way paraphenoltolylamine, $OH\cdot C_6H_4\cdot NH\cdot C_6H_4Me$, may be made to yield the compound $C_6H_4<\begin{matrix}N(C_6H_4Me)\\O\text{————}\end{matrix}>$, which much resembles its homologue above described. On evaporation of a solution in light petroleum, crystals of a deep red colour and dark green lustre are formed. These melt at 70°, and likewise yield quinone on boiling with water. The author has also isolated the compound $C_6H_4<\begin{matrix}N(C_6H_4Me)\\N(C_6H_4Me)\end{matrix}>$, from paraditolylamidobenzene, $C_6H_4(NH\cdot C_6H_4Me)_2$. G. T. M.

Nitrogenous Quinone-derivatives. By R. Nietzki and A. W. Schmidt (*Ber.*, **21**, 1850—1856).—*Diamidodihydroxyquinone*, $C_6H_6N_2O_4$, is formed when sodium nitrite is added to a solution of diamidotetrahydroxybenzene hydrochloride, or when a solution of the latter is exposed to the air after neutralising the hydrochloric acid present; after adding alcohol the product is precipitated in reddish-brown needles by means of aniline or sodium acetate. This compound is also produced by the action of phenanthraquinone on a glacial acetic acid solution of diamidotetrahydroxybenzene hydrochloride or by partially oxidising the latter with ferric chloride. After drying at 100°, it contains water which is, however, evolved at 150°. When treated with concentrated hydrochloric acid, it yields a yellowish, crystalline *hydrochloride* which is decomposed by water. When heated with anhydrous sodium acetate and acetic anhydride, it gives a reddish-brown, crystalline compound which is probably a mixture of several acetyl-derivatives.

When a solution of the reddish sodium salt obtained by dissolving hexacetyldiamidotetrahydroxybenzene in soda (Nietzki and Benckiser, Abstr., 1885, 779), or of the sodium salt of diacetyldiamidohydroquinone (Nietzki and Preusser, Abstr., 1886, 1024) is treated with hydrochloric acid, *diacetyldiamidodihydroxyquinone*, $C_6(OH)_2O_2(NHAc)_2$, is precipitated in brownish-red needles.

Diacetyldiamidotetrahydroxybenzene, $C_6(OH)_4(NHAc)_2$, is formed when the preceding compound is treated with stannous chloride and dilute hydrochloric acid. It crystallises from alcohol in colourless needles, and is quickly converted into the quinone when its alkaline solution is exposed to the air.

The characteristic reddish needles of the sodium salt of diacetyldiamidodihydroxyquinone separate when the reddish-brown product obtained by heating diamidodihydroxyquinone with sodium acetate and acetic anhydride is dissolved in soda, and the solution kept for some time. The constitution of diamidodihydroxyquinone is therefore $[O_2 : (NH_2)_2 : (OH)_2 = 1 : 4 : 2 : 5 : 3 : 6]$.

Diquinoylimide, $C_6O_4(NH)_2 + 5H_2O$ $[(NH)_2 = 3 : 6]$, is obtained as a bye-product in the preparation of triquinoyl from diamidotetrahydroxybenzene. It is a colourless compound which is insoluble in ordinary solvents, and is decomposed with evolution of gas by boiling with water. It is converted into diamidotetrahydroxybenzene when treated with stannous chloride and hydrochloric acid.

Tetrahydroxyquinoneanilide, $C_6O(OH)_4{:}NPh$ $[O : (NPh) = 1 : 4]$, is obtained when hexahydroxybenzene is dissolved in dilute alcohol, aniline added, and the solution exposed to the air. It crystallises in shining, golden leaves which have a carmine-red colour by transmitted light, and are very sparingly soluble in all neutral solvents, but soluble in alkalis with decomposition.

The compound $C_6(OH)_2O_2{:}NPh,NH_2Ph$ is obtained in red needles with a superficial green tinge when tetrahydroxyquinone is treated with aniline. *Aniline rhodizonate*, $C_6H_2O_6(NH_2Ph)_2$, separates in red needles when a dilute hydrochloric acid solution of sodium rhodizonate is treated with aniline.

Croconamic acid (Nietzki and Benckiser, Abstr., 1886, 540) forms a *barium* salt which crystallises in sparingly soluble, yellow leaves, and has the composition $C_5HNO_4Ba + 4H_2O$ when dried at 120°. Croconamic acid has therefore the constitution $C_5O_2(OH)_2{:}NH$.

F. S. K.

Tautomerism of Thiocarbamides. By F. FOERSTER (*Ber.*, **21**, 1857—1873).—*Methyl anisylimidoanisylthiocarbamate*,

$$C_7H_7O{\cdot}N{:}C(SMe){\cdot}NH{\cdot}C_7H_7O,$$

prepared by treating dianisylthiocarbamide with methyl iodide, crystallises from alcohol in small, white prisms, melts at 87°, is readily soluble in hot alcohol and ether, moderately soluble in cold alcohol, and almost insoluble in water. It is readily soluble in sulphuric acid, hydrochloric acid, and acetic acid, moderately soluble in nitric acid and hydrobromic acid, and still less soluble in hydriodic acid. The *hydrochloride*, $C_{16}H_{18}N_2O_2S,HCl$, crystallises in white leaves. The *platinochloride*, $(C_{16}H_{18}N_2O_2S)_2,H_2PtCl_6$, crystallises in

small, short prisms which are more readily soluble in alcohol than in water. The *aurochloride* is amorphous, and melts under water. When the base is distilled, methylmercaptan and carbodianisylimide are formed. Alkalis decompose it into methylmercaptan and dianisylcarbamide. Treated with alcoholic ammonia, it yields methylmercaptan and *dianisylguanidine*, $C_7H_7O{\cdot}N{:}C(NH_2){\cdot}NH{\cdot}C_7H_7O$, and with alcoholic anisidine it gives methylmercaptan and trianisylguanidine, $C_7H_7O{\cdot}N{:}C(NH{\cdot}C_7H_7O)_2$.

Methyl anisyldithiocarbamate, $SMe{\cdot}CS{\cdot}NHC_7H_7O$, and anisylthiocarbamide are obtained when the base is digested with carbon bisulphide; the former compound is soluble in very dilute alkalis, and is reprecipitated by hydrochloric acid; it is decomposed by concentrated alkalis, and yields anisylthiocarbamide and methylmercaptan when heated.

Ethyl anisylimidoanisylthiocarbamate, $C_{17}H_{20}N_2O_2S$, prepared by treating dianisylthiocarbamide with ethyl iodide, melts at 82·5°, and resembles the corresponding methyl base. The *hydriodide*,

$$C_{17}H_{20}N_2O_2S,HI,$$

crystallises in large, shining rhombohedra which melt at 162—163° with decomposition, and are insoluble in ether. The *platinochloride*, $(C_{17}H_{20}N_2O_2S)_2,H_2PtCl_6$, is similar to the salt of the methyl base.

Propyl anisylimidoanisylthiocarbamate, $C_{18}H_{22}N_2O_2S$, prepared similarly, crystallises in shining leaves melting at 58°. This base and its *platinochloride* closely resemble the corresponding methyl- and ethyl-derivatives.

Ethylene anisylimidoanisylthiocarbamate, $C_7H_7ON{:}C{<}\begin{matrix}N(C_7H_7O)\\S{\cdot}CH_2{\cdot}CH_2\end{matrix}{>}$, prepared by heating dianisylthiocarbamide with ethylene bromide, crystallises in white prisms, melts at 128°, is readily soluble in benzene and hot alcohol, less readily soluble in light petroleum, and insoluble in water. The *hydrochloride* and *sulphate* are readily soluble in water, the *hydrobromide* and *hydriodide* are less readily soluble and the *picrate* is only sparingly soluble. The *platinochloride*, $(C_{17}H_{18}N_2O_2S)_2,H_2PtCl_6$, is only stable in the dry state.

Ethylene anisyldithiocarbamate, $CS{<}\begin{matrix}N(C_7H_7O)\\S{\cdot}CH_2{\cdot}CH_2\end{matrix}{>}$, prepared by heating the preceding compound with carbon bisulphide, is a neutral substance which crystallises from alcohol in hard prisms, and from benzene, in which it is readily soluble, in large, transparent crystals melting at 136°, and rather readily soluble in hot alcohol and ether. It is with difficulty converted into the monothio-derivative by mercuric oxide, silver oxide, or silver nitrate, and it combines with methyl iodide to form a crystalline compound, $C_{10}H_{11}NOS_2,MeI$, which melts at about 141°, is soluble in water, alcohol, and benzene, but insoluble in ether. When digested with anisidine, it gives mercaptan and ethylene anisylimidoanisylthiocarbamate.

Ethylene anisylthiocarbamate, $CO{<}\begin{matrix}N(C_7H_7O)\\S{\cdot}CH_2{\cdot}CH_2\end{matrix}{>}$, prepared by heating the dithiocarbamate with alcohol and chloracetic acid, crystallises

in shining leaves, melts at 116°, and is readily soluble in alcohol, ether, and benzene, but insoluble in water.

Dianisylthiohydantoïn, $C_7H_7ON{:}C{<}^{N(C_7H_7O)}_{S\cdot CH_2\cdot CO}{>}$, is obtained when dianisylthiocarbamide is heated with chloracetic acid. It crystallises from glacial acetic acid in bright yellow needles, melts at 190°, is readily soluble in benzene and glacial acetic acid, sparingly soluble in hot alcohol, but insoluble in water or cold alcohol.

Anisyl phenylthiocarbamide, $NHPh\cdot CS\cdot NH\cdot C_7H_7O$, produced by treating phenylthiocarbimide with anisidine, or by the action of anisylthiocarbimide on aniline, crystallises in white prisms melting at 127°. When digested with ethylene bromide, it yields a resinous product from which a crystalline base, $NPh{:}C{<}^{N(C_7H_7O)}_{S\cdot CH_2\cdot CH_2}{>}$, melting at 143°, was isolated. Two isomeric bases are formed when α-naphthylphenylthiocarbamide is treated with ethylene bromide. The one is sparingly soluble in alcohol, crystallises in needles melting at 184·5°, and yields phenylthiocarbimide and ethylene α-naphthyldithiocarbamate when treated with carbon bisulphide; it can also be obtained by the action of aniline on the additive product of methyl iodide and ethylene α-naphthyldithiocarbamate; its constitution is, therefore, $NPh{:}C{<}^{N(C_{10}H_7)}_{-S\cdot C_2H_4-}{>}$. The other is more readily soluble in alcohol, and crystallises in shining leaves melting at 129—130°; it is, however, impure, as it yields the decomposition products of both the theoretical bases when treated with carbon bisulphide. By acting on the additive product of methyl iodide and ethylene phenyldithiocarbamate with α-naphthylamine, the pure compound melting at 134·5° is obtained; it yields naphthylthiocarbimide and ethylene phenyldithiocarbamate when treated with carbon bisulphide. Its constitution is, therefore, $C_{10}H_7\cdot N{:}C{<}^{-NPh-}_{S\cdot C_2H_4}{>}$.

A base, $NPh{:}C(SMe)\cdot NHC_7H_7O$, which crystallises in long, white, silky needles melting at 80°, is obtained by the action of methyl iodide on anisylphenylthiocarbamide. It yields phenylthiocarbimide when treated with carbon bisulphide. The base

$$C_{10}H_7N{:}C(SMe)\cdot NHPh,$$

obtained from methyl iodide and α-naphthylphenylthiocarbamide, crystallises in small, white needles which melt at 96°.

Phenyltrihydrothiazole, $NPh{<}^{C_2H_4}_{CH_2}{>}S$, is obtained by reducing an alcoholic solution of ethylene phenyldithiocarbamate with zinc and hydrochloric acid. It is an oily base which is readily soluble in alcohol and ether, but insoluble in water; it is volatile with steam, and has a slight smell resembling that of conine.

Trimethylene phenylimidophenylthiocarbamate, $NPh{<}^{C(NPh)}_{-C_3H_6-}{>}S$, formed by the action of trimethylene bromide on diphenylthiocarbamide, crystallises in white, silky needles, melts at 123°, and behaves similarly to the ethylene base.

Trimethyleneanisylimidoanisylthiocarbamate,

$$C_7H_7ON<\begin{matrix}C(C_7H_7ON)\\ \text{---}C_3H_6\text{---}\end{matrix}>S,$$

prepared similarly, crystallises from alcohol in well-defined prisms melting at 113—114°.

Methylene phenylimidophenylthiocarbamate, $NPh<\begin{matrix}C(NPh)\\ \text{---}CH_2\text{---}\end{matrix}>S$, prepared by acting on diphenylthiocarbamide with methylene bromide, crystallises in long needles, melts at 68°, and is very readily soluble in alcohol. The *platinochloride*, $(C_{14}H_{12}N_2S)_2,H_2PtCl_6$, is crystalline.

F. S. K.

Guanidines and Guanidine Cyanides. By L. Hirsch (*Chem. Centr.*, 1888, 624—625).—If cyanogen gas is led into an ethereal solution of nitrodiphenylguanidine, prepared by desulphurising nitrodiphenylthiocarbamide with ammonia, *nitrodiphenylguanidine dicyanide*, $NH:C<\begin{matrix}NPh\text{---------}C(NH)\\ N(C_6H_4\cdot NO_2)\cdot C(NH)\end{matrix}>$, is formed, which decomposes on heating. Dilute hydrochloric acid changes it into *nitrodiphenyloxalylguanidine*, $NH:C<\begin{matrix}NPh\text{---------}CO\\ N(C_6H_4\cdot NO_2)\cdot CO\end{matrix}>$, melting at 168°. If an alcoholic solution of this compound is heated with concentrated hydrochloric acid, *nitrodiphenylparabanic acid*, $CO<\begin{matrix}NPh\text{---------}CO\\ N(C_6H_4\cdot NO_2)CO\end{matrix}>$, is formed.

Attempts to prepare α-cyanogen additive products by acting on nitrotriphenylguanidine with cyanogen gas failed, nor were any definite substances obtained by reducing the above nitroguanidine with zinc and hydrochloric acid.

β-Nitrotriphenylguanidine dicyanide, $C_{21}H_{16}N_6O_2$, can be obtained by the action of an alcoholic solution of aniline hydrochloride on the cyanogen additive products of the corresponding diphenylguanidine. Reduction of this compound with zinc and hydrochloric acid produces a complete decomposition, aniline, ammonium chloride, and diphenylguanidine being formed.

Bromoguanidine is obtained by acting on phenyl thiocarbimide with parabromaniline, and desulphurising with ammonia. If an ethereal solution of bromodiphenylguanidine melting at 147° is treated with cyanogen gas, *bromodiphenylguanidine dicyanide* is formed, which is converted by acids, even in the cold, into bromodiphenyloxalylguanidine melting at 156°. These two compounds when treated with concentrated hydrochloric acid give bromodiphenylparabanic acid, melting at 194°.

Attempts were made to desulphurise diphenylthiocarbamide with diamines. Phenylenediamine and tolnylenediamine did not produce any characteristic product. Benzidine formed dibenzylthiocarbamide.

When diphenylguanidine dicyanide was treated with amines of the aromatic series, no definite products were formed, only a slightly fluorescent, red coloration. Diphenylamine and cumidine hydro-

chloride react on one another, but no pure product could be separated.

Phenylenediamine and diphenylguanidine dicyanide, both in alcoholic solution, form a product yielding a red hydrochloride. This salt is so insoluble in the various solvents that it is difficult to obtain it pure. Benzidine hydrochloride acts similarly.

If phenyl-β-naphthylthiocarbamide is desulphurised with aniline, triphenylguanidine is formed. J. P. L.

Benzazimide. By H. Finger (*J. pr. Chem.* [2], **37**, 431—445).—The author applies the name *benzazimide* to the compound $C_7H_5N_3O$, obtained by the action of nitrous acid on orthamidobenzamide, and previously described by Weddige and himself (Abstr., 1887, 667).

Orthamidobenzethylamide, $NH_2{\cdot}C_6H_4{\cdot}CONHEt$, is obtained by the action of an alcoholic solution of ethylamine on isatoic acid. It crystallises from benzene in leaflets or feathery needles melting at 104—105°. When it is dissolved in hydrochloric or nitric acid and the calculated quantity of sodium nitrite added, *ethyl benzazimide*, $C_6H_4<^{CO{\cdot}NEt}_{N:N}>$, separates, at first in oily drops and afterwards in needles, soluble in ether, alcohol, chloroform, and hot water, melting at 70°, and of an agreeable, ethereal odour.

Benzazimide is decomposed by hot, strong alkalis into orthamidobenzoic acid and nitrogen. Strong hydrochloric acid dissolves both benzazimide and methylbenzazimide in the cold without change, but when heated with either of them in a sealed tube at 120—130°, nitrogen and a little carbonic anhydride are evolved, a crystalline mass is formed, consisting of chlorosalicylic acid and a little salicylic acid, and ammonia or methylamine is found in solution.

Orthethamidobenzamide, $NEtH{\cdot}C_6H_4{\cdot}CONH_2$ (compare Abstr., 1887, 1044), is obtained when orthamidobenzamide is heated with the calculated quantity of alcoholic ethyl iodide in a sealed tube at 100°. The contents are allowed to remain in the cold for 12 hours, then evaporated to a syrup, and treated with ammonia; the oil thus precipitated gradually solidifies, and is recrystallised from hot water; it melts at 128—129°.

Orthonitrosomethamidobenzamide, $NO{\cdot}NMe{\cdot}C_6H_4{\cdot}CONH_2$, is obtained by the action of nitrous acid on orthomethamidobenzamide, as white needles, which crystallise from hot alcohol in shining, pale-yellow prisms, and from hot water in shining leaflets; it melts at 149°. The *nitrosoethyl*-derivative crystallises from hot water in pale-yellow, prismatic needles melting at 110°. Both of these give Liebermann's colour reaction.

Alcoholic hydrogen chloride in the cold converts orthonitrosomethamidobenzamide into orthomethamidobenzamide and nitrous acid. Aqueous hydrogen chloride produces a mass which gives an intensely green hydrochloride when treated with pure water; this crystallises in green needles, and is not the paranitroso-compound (compare Abstr., 1887, 244, 729).

The foregoing reactions confirm the structural formula already given for benzazimide (Abstr., 1887, 667). A. G. B.

Hydrazines. By C. WILLGERODT (*J. pr. Chem.* [2], **37**, 449—454).—Two symmetrical dinitrophenyl-phenylhydrazines seem to be formed by the action of an alcoholic solution of symmetrical meta-dinitrochlorbenzene on phenylhydrazine; in the cold a solid is formed, but in the warm from the same mixture a syrup was obtained on several occasions. If further researches should prove the existence of the syrupy modification, it must be a geometrical isomeride of the solid. The question will soon be set at rest, as the author has obtained the syrupy compound alone by employing ethereal in place of alcoholic solutions of the reacting substances.

Nitrobromophenyl-phenylhydrazine, $NO_2 \cdot C_6H_3Br \cdot NH \cdot NHPh$ [4 : 1 : 3], is obtained by mixing symmetrical orthodinitrobromobenzene with phenylhydrazine in cold alcoholic solution; it forms red needles melting at 165°. When it is heated in glacial acetic acid, *nitrosobromazobenzene*, $NO \cdot C_6H_3Br \cdot N{:}NPh$, is obtained as white needles melting at 124°, and when it is oxidised with mercuric oxide, *nitrobromazobenzene* is produced in red needles melting at 103·5°. A syrup is obtained by the action of symmetrical orthodinitrobromobenzene on phenylhydrazine in cold ethereal solution, which is also converted into nitrosobromazobenzene by glacial acetic acid, and into nitrobromazobenzene by mercuric oxide. The behaviour of tolyl- and other hydrazines, as well as the whole subject, is still undergoing investigation.

A. G. B.

Indamines and Indophenols. By R. NIETZKI and R. OTTO (*Ber.*, **21**, 1736—1745).—Gallocyanin (Koechlin, Abstr., 1883, 796) is very sparingly soluble in water, alcohol, and glacial acetic acid, readily in aniline, with formation of an anilide; alkalis and alkaline carbonates dissolve it readily with reddish colour. It has the composition expressed by the formula $C_{15}H_{12}N_2O_5$. The *anilide*, $C_{27}H_{24}N_4O_4$, already mentioned, crystallises in long needles with a greenish lustre, yields crystalline but unstable salts with acids, and is decomposed by alkalis into gallocyanin and aniline.

The dye known as "prune," obtained from methyl gallate and nitrosodimethylaniline, has the formula $C_{15}H_{12}MeN_2O_5$. It is a stronger base than gallocyanin, and does not yield an anilide when crystallised from aniline, but an additive compound, $C_{16}H_{14}N_2O_5,C_6H_7N$.

The constitution of gallocyanin is probably—

$$COOH \cdot C_6H(OH)_2 < \begin{matrix} N \\ O \end{matrix} > C_6H_3 \cdot NMe_2 \cdot R.$$

A *diacetyl-derivative*, $C_{20}H_{18}N_2O_7$, was prepared, which crystallises in small, greenish needles.

Quinonedichlorimide reacts with β-naphthol in alcoholic solution with formation of a reddish-violet dye, which is readily precipitated as the zincochloride. When this is dissolved in water and treated with nitric acid, the *nitrate*, $C_{16}H_{10}N_2O,HNO_3$, separates. The blue dye prepared by Meldola (Abstr., 1880, 162), by the action of nitrosodimethylaniline on β-naphthol, is without doubt the dimethyl-derivative of this substance, and must therefore have the formula $C_{18}H_{14}N_2O$ (instead of $C_{18}H_{16}N_2O$).

N. H. M.

Phthalimide. By E. Bamberger and R. Müller (*Ber.*, **21**, 1888—1892).—When phthalimide in portions of 6 grams is dissolved in 150 grams of amyl alcohol and treated with 12 grams of sodium, a substance, termed by the author *orthotolubenzylamine*,

$$C_6H_4Me\cdot CH_2\cdot NH_2,$$

is obtained as the chief product. This is identical with Strassmann's orthoxylylamine (this vol., p. 474). It boils at 201° under 718 mm. pressure, and at 125° under 105 mm. pressure; it is sparingly soluble in cold water. When exposed to air it solidifies, forming lustrous needles of the carbonate. The *picrate* forms very lustrous, branched needles, which decompose at 205—220° without melting.

A second, more feeble base, probably of the formula

$$C_6H_4<\begin{matrix}CH_2\\CH_2\end{matrix}>NH,$$

is also formed in the reduction of phthalimide. It is a clear liquid, of an odour resembling that of quinoline and of conine; the *hydrochloride* forms white plates. The *platinochloride* crystallises in lustrous, orange-coloured needles. N. H. M.

Action of Sulphur on Benzaldehyde. By A. Gazzarrini (*Chem. Centr.*, 1888, 781—782, from *Atti Acad. Sci. Toscano*, **6**, 64—65).—Sulphur does not readily act on benzaldehyde, for when the two substances are heated together in a sealed tube at 160°, no visible action takes place even after eight hours. On heating the tube at 180° for eight hours, and finally at 190° for 15 hours, the two substances do react. Hydrogen sulphide occurs among the gaseous products. The solid products of the reaction are benzoic acid and a substance containing sulphur, which crystallises in silky, prismatic needles.

All attempts to separate this latter substance from the benzoic acid either by neutralising the benzoic acid with sodium carbonate or barium hydroxide, or by fractional distillation failed, only benzoic acid and a metallic sulphide or hydrogen sulphide being obtained. This substance is easily soluble in ether and undergoes decomposition very easily in air and in contact with water, especially in the warm.

J. P. L.

Combination of Mannitol Anhydride with Benzaldehyde. By J. Meunier (*Compt. rend.*, **106**, 1425—1426).—10 grams of mannitol, 20 grams of absolute alcohol, and 5 grams of fused zinc chloride are mixed together, treated with dry hydrogen chloride, and allowed to remain for two days. The liquid portion is then decanted from unaltered mannitol, and mixed with one-fifth the quantity of benzaldehyde. There is a rapid and abundant formation of acicular crystals of the compound $C_6H_8O_3(C_7H_6O)_2$. These are washed with water and alcohol and recrystallised from benzene. They are insoluble in water, slightly soluble in alcohol, and much more soluble in boiling benzene, melt at 207° with slight decomposition, and tend to remain in superfusion. The compound is not affected even by boiling alcoholic solutions of the caustic alkalis, but is readily decomposed by acids with liberation of benzaldehyde. C. H. B.

Action of Chloracetonitrile on Benzene in presence of Aluminium Chloride. By P. GENVRESSE (*Bull. Soc. Chim.*, **49**, 579—581).—The author heated chloracetonitrile (1 mol.) and benzene (3 mols.), together with aluminium chloride (1½ mols.), for about 12 hours and poured the cold mixture into water. The reaction-product, mixed with the excess of benzene and chloracetonitrile, separated, and was fractioned. Orthotoluic acid, benzoic acid, and orthotolunitrile were isolated from the mixture, but only a small portion of the chloracetonitrile had been acted on. F. S. K.

Methyl Benzoylcyanacetate and Cyanacetophenone. By L. BARTHE (*Compt. rend.*, **106**, 1416—1418).—Benzoic chloride dissolved in ether is added gradually to a well-cooled solution of methyl-cyanacetate and sodium methoxide in methyl alcohol. The liquid is heated to complete the reaction, and extracted with ether and a solution of sodium carbonate. The aqueous liquid is removed, filtered, neutralised with sulphuric acid, and the precipitate recrystallised from alcohol. *Methyl benzoylcyanacetate* forms long, transparent, prismatic crystals which melt at 74°, and are soluble in ether and alcohol. It gives a red colour with ferric salts, and the alcoholic solution has an acid reaction. The sodium-derivative,

$$CN\cdot CPhNa\cdot COOMe,$$

is obtained by dissolving the compound in alcohol, adding sodium carbonate to the hot solution, and allowing the neutral liquid to evaporate spontaneously. It forms hard, white, transparent crystals which decompose at 120—125°, and are soluble in water or alcohol. The barium-derivative is obtained in a similar manner, and crystallises with 1 mol. H_2O.

When methyl benzoylcyanacetate is boiled with water, carbonic anhydride is evolved; and when the liquid is cooled, it deposits cyanacetophenone, $COPh\cdot CH_2\cdot CN$, in long, silky, white needles which melt at 81·5°. C. H. B.

Synthesis of Toluoylpropionic Acid. By E. BURCKER (*Bull. Soc. Chim.*, **49**, 448—450).—*Toluoylpropionic acid*, $C_7H_7\cdot CO\cdot C_2H_4\cdot COOH$, is formed by the action of succinic anhydride on toluene in presence of anhydrous aluminium chloride. It crystallises from boiling water in laminæ or slender needles, is readily soluble in ether, alcohol, benzene, chloroform, toluene, and boiling water, but insoluble in cold water; it melts at 120°. When heated slightly above this temperature, it turns red, and retains the colour when cold; at a higher temperature it is decomposed. The *potassium* and *sodium* salts are readily soluble in water. The *barium* salt, $(C_{11}H_{11}O_3)_2Ba + 4H_2O$, is soluble in boiling water, from which it separates in mamelons which lose the whole of their water at 110°. The *silver* salt, $C_{11}H_{11}O_3Ag$, crystallises in small needles and is insoluble in water. The *cobalt*, *nickel*, and *copper* salts are crystalline. F. S. K.

Dianilidosuccinic Acid. By J. GORODETZKY and C. HELL (*Ber.*, **21**, 1795—1801).—*Ethyl dianilidosuccinate*, $C_{20}H_{24}N_2O_4$, is prepared by

heating pure ethyl dibromosuccinate with aniline (4 mols.) on a water-bath. The product is extracted with ether, the ether distilled off, and the residue again heated on the water-bath, when a further separation takes place. The crystals are digested with water made slightly acid with hydrochloric acid to remove the aniline salt and recrystallised from alcohol. It forms slender needles of a silky lustre, melts at 150°, dissolves readily in chloroform and hot alcohol, sparingly in ether, benzene, and light petroleum; it also dissolves in strong sulphuric and hydrochloric acids, but is precipitated by water.

Dianilidosuccinic acid, $C_{16}H_{16}N_2O_4$, is obtained by heating the ethyl salt with alcoholic potash at 60—70°; on adding acid to the solution of the potassium salt, it separates as a crystalline precipitate. It is a white, very light powder, almost insoluble in water, light petroleum, and benzene, more soluble in alcohol, warm ether, and chloroform. It melts at 190° with decomposition. The alkali and barium salts are soluble in water; the other salts are almost insoluble. The *sodium salt* crystallises from hot water in lustrous spear-heads; the *potassium salt* is very readily soluble in water, and is precipitated by alcohol in cubes; the *calcium*, *lead*, and *silver salts* are also described.

Ethyl hexabromodianilidosuccinate, $C_2H_2(NH\cdot C_6H_2Br_3)_2(COOEt)_2$, prepared by adding bromine to a solution of ethyl dianilidosuccinate in chloroform, melts at 98—99° when crystallised from light petroleum, and at 103—104° when crystallised from alcohol; it is readily soluble in ether, chloroform, benzene, hot light petroleum, and hot alcohol, insoluble in water. The *free acid* is a white, amorphous substance, melts at 230° with decomposition, and is very sparingly soluble in the usual solvents, insoluble in water. The *potassium salt* forms white, lustrous needles rather sparingly soluble in cold water; the *sodium salt* is much more sparingly soluble in water; the *barium*, *silver*, and *ammonium salts* also dissolve very sparingly. The acid as well as its salts are much more stable than dianilidosuccinic acid.

N. H. M.

Chlorides of Bibasic Acids. By V. Auger (*Bull. Soc. Chim.*, **49**, 345—352).—*Succinophenone*, $COPh\cdot C_2H_4\cdot COPh$, and *diphenylbutyrolactone*, $C_2H_4<\begin{matrix}CPh_2\\CO\end{matrix}>O$, are produced when aluminium chloride is added gradually to a mixture of succinic chloride and a large excess of benzene at 25—30°. The reaction product is crystallised from boiling alcohol, when succinophenone separates first in needles; as soon as small leaves begin to deposit, the solution is decanted; the compounds thus separated are recrystallised from alcohol. Succinophenone melts at 134°, and is insoluble in warm concentrated potash.

Diphenylbutyrolactone melts at 90°, and is readily soluble in warm potash; when this solution is acidified, *γ-diphenyl-γ-hydroxybutyric* acid is precipitated in white flocks which are insoluble in water, but soluble in alcohol. This acid separates from alcohol in large, tabular crystals; when heated at 145,° it melts and is reconverted into the lactone with evolution of 1 mol. H_2O. The *barium* salt, $(OH\cdot CPh_2\cdot C_2H_4\cdot COO)_2Ba$, crystallises in lustrous, stellate forms which are soluble in water.

From these experiments it follows that succinic chloride is a mixture of the two theoretically possible chlorides.

When succinic chloride is added drop by drop to well-cooled ammonia, succinamide is deposited; the filtered solution is evaporated over sulphuric acid, and the residue extracted with alcohol. A heavy, yellow oil is precipitated when the alcoholic extract is mixed with ether, and this oil solidifies to a granular mass after some weeks. It has the composition $C_2H_4(CONH_2)_2$, is exceedingly hygroscopic, and melts at about 90°. When treated with acids, it yields succinic acid. Its aqueous solution is precipitated by silver nitrate with formation of a *silver* salt, $C_2H_4{:}C_2O_2{:}NAg$, which is almost totally insoluble in water.

Phthalamic acid, $COOH{\cdot}C_6H_4{\cdot}CONH_2$, is prepared by adding hydrochloric acid (1 mol.) to an aqueous solution of ammonium phthalamate; the last named compound is obtained when an ammoniacal solution of phthalic anhydride is crystallised. Phthalamic acid crystallises in small, acute prisms which are sparingly soluble in water. When heated at about 140° it melts, loses water, and is converted into phthalimide. Treated with an excess of hydrochloric acid it is decomposed after some time with formation of phthalic acid. The *silver* salt is almost totally insoluble in water.

The compound, $C_6H_4{:}C_2O_2(NH_2)_2$, is formed, with evolution of heat, when phthalic chloride is added gradually to ammonia. It crystallises from cold alcohol in long, silky needles, and from hot alcohol in long, transparent prisms which are readily soluble in water. When heated, it softens at about 90°, and melts at about 140—160° with evolution of ammonia, but solidifies immediately and does not melt again until 232°, the melting point of phthalimide. When silver nitrate is added to an aqueous solution, the salt, $C_6H_4{:}C_2O_2{:}NAg$, is precipitated in lustrous scales which are almost totally insoluble in cold water.

The free acid, $C_6H_4{:}C_2O_2{:}NH$, separates as a curdy precipitate when hydrochloric acid (1 mol.) is added to a dilute solution of the reaction product of phthalic chloride and ammonia. It crystallises from boiling alcohol in long needles which are only sparingly soluble in water. When heated, it melts at about 145°, and is converted into phthalimide. It has an acid reaction, and its aqueous solution decomposes barium carbonate in the cold. If the curdy precipitate mentioned above is crystallised from boiling water, large, transparent needles having the composition $C_{16}H_9O_5N$ are obtained. This compound is very readily soluble in water, to which it imparts an acid reaction. When heated at 145—150° it partially melts, but solidifies again, and yields a mixture of phthalic anhydride and phthalimide on further heating.

From these results it follows that phthalic chloride has the asymmetrical formula.

Phthalimide has a distinctly acid reaction, and its boiling aqueous solution decomposes barium carbonate with formation of a barium salt which is soluble in water. When heated with ammonia, it yields ammonium phthalimide in the form of a powder which is reconverted into phthalimide by acids. When the ammoniacal solution is boiled with the precipitate for some time, the latter is completely dissolved,

and an addition of hydrochloric acid does not precipitate phthalimide; the evaporated solution deposits small prisms, which are soluble in water and have an acid reaction. F. S. K.

Constitution of Pyranilpyroïc Acid, Pyranilpyroïn Lactone, and Anilsuccinic Acid. By A. Reissert (*Ber.*, **21**, 1942—1947; compare this vol., p. 694).—An answer to Anschütz (*Annalen*, **246**, 115).

Desmotropy. By A. Hantzsch and F. Herrmann (*Ber.*, **21**, 1754—1758; compare *Ber.*, **20**, 2800).—Ethyl dichlorohydroquinonedicarboxylate dissolves in benzene, chloroform, and in strong sulphuric acid with intense greenish-yellow coloration, whilst the alcoholic solution, like the substance itself, is colourless. It is shown that the alcoholic solution contains the salt as an alcoholate which separates in large crystals of the formula $C_6H_2O_2Cl_2(COOEt)_2,2EtOH$, whilst the salt, when dissolved in solvents which do not combine with it, undergoes the same change as when it is fused, being converted into the coloured modification—ethyl dichloroquinonehydrodicarboxylate. N. H. M.

Desmotropic Derivatives of Ethyl Succinosuccinate. By M. Böniger (*Ber.*, **21**, 1758—1765).—The ethyl salt, $C_6H_2Br_2O_2(COOEt)_2$, obtained by Herrmann (Abstr., 1886, 1028) by the action of bromine vapour on ethyl quinonedihydroparadicarboxylate, can also be prepared, directly from ethyl succinosuccinate by the action of dry bromine. It is probably ethyl dibromohydroquinonedicarboxylate,

$$[Br_2 : (OH)_2 : (COOEt)_2 = 2 : 5 : 3 : 6 : 1 : 4].$$

It melts at 157°, and when sublimed in a slow stream of air it undergoes an intramolecular change, and the compound *ethyl dibromoquinonehydrodicarboxylate*, $[Br_2 : O_2 : (H,COOEt)_2 = 2 : 5 : 3 : 6 : 1 : 4]$, is obtained in yellow, tabular crystals, which, when kept at the ordinary temperature, recover their original form and colour. The yellow crystals dissolve in benzene, yielding a yellow solution.

When sodium dibromohydroquinonedicarboxylate is dissolved in a little hot water, and carefully acidified, *dibromoquinonehydrodicarboxylic acid* separates as a lemon-yellow, microcrystalline powder. This is very unstable, and becomes gradually lighter, until it is finally white. The stable modification, *dibromohydroquinonedicarboxylic acid*, so obtained, is almost insoluble in water, alcohol, and ether.

Ethyl dibromoquinonedicarboxylate (Herrmann, *loc. cit.*) melts at 221°. When dissolved in benzene, and treated with ammonia, it is converted into the diamido-derivative.

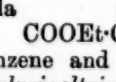
Ethyl diamidoquinonehydrocarboxylate, $[O_2 : (NH_2)_2 : (H,COOEt) = 3 : 6 : 2 : 5 : 1 : 4]$, obtained by reducing the above amido-salt suspended in alcohol with tin and hydrochloric acid, forms splendid red needles. It is unstable, and readily becomes changed into ethyl diamidoquinonedicarboxylate. The free acid could not be prepared. The *hydrochloride* crystallises in long, lemon-coloured needles, and is decomposed by water with intense red colour. The *stannochloride*,

$C_{12}H_{16}N_2O_6,SnCl_4 + 2H_2O$, forms long, yellow needles. When the hydrochloride is heated with acetic anhydride, the *diacetate* of *ethyl diamidohydroquinonedicarboxylate*, $C_6(OH)_2(NHAc)_2(COOEt)_2$, is formed; this crystallises from glacial acetic acid in stellate groups of colourless, lustrous needles, melting at 236°, without decomposition. The *tetracetyl-derivative*, $C_6(OAc)_2(NHAc)_2(COOEt)_2$, obtained by boiling the diacetyl-compound for a long time with an excess of acetic anhydride, is a colourless, microcrystalline substance, melting at 206°. Both acetates are soluble in alkalis.

Diimidoquinonehydrodicarboxylic acid, [(H,COOH)$_2$: (NH)$_2$ = 1 : 4 : 3 : 6], is prepared by saponifying v. Baeyer's so-called ethyl diamidonaphthalate (Abstr., 1886, 445), and forms greenish-yellow, prismatic crystals, almost insoluble in the usual solvents. The *hydrochloride*, $C_6H_8N_2O_4,2HCl$, crystallises in colourless plates, which decompose when air-dry, giving off hydrogen chloride. The free acid corresponds with the quinonehydrodicarboxylic acid, whilst in taking up acid it is converted into salts of the colourless modification, diamidoterephthalic acid. N. H. M.

Isomeride of Orthophenylphthalidecarboxylic Acid. By P. Jouillard (*Chem. Centr.*, 1888, 520, from *Arch. Sc. Phys. Nat. Genève*, **19**, 121—131).—Phenylacetic acid acts on phthalic anhydride with the formation of benzylidenephthalyl, which reacts with water to form benzylidenephthalylcarboxylic acid. The acid forms an acetoxime melting at 113—114°. When oxidised with potassium manganate, *benzilcarboxylic acid*, melting at 137—138°, is formed. The methyl salt melts at 117—118°, the ethyl salt at 70·5—71·5°.

Benzilcarboxylic acid decomposes with potash, like diphthalic acid, phenylphthalidecarboxylic acid being formed; this crystallises with 1 mol. H_2O, which it loses at 100°; at the same time it undergoes decomposition at this temperature, carbonic anhydride being given off, and the lactone of a benzhydrylcarboxylic acid being formed, which melts at 115°. On oxidation it is converted into orthobenzoylbenzoic acid, melting at 85—87°. J. P. L.

Ethyl Phloroglucinoltricarboxylate. By O. Bally (*Ber.*, **21**, 1766—1771).—In the preparation of ethyl phloroglucinoltricarboxylate by v. Baeyer's method (Abstr., 1886, 223), an anhydride of the formula  is formed. This is almost insoluble in benzene and alcohol, and melts at 168—170°. *Ethyl triacetylphloroglucinoltricarboxylate*, $C_6(OAc)_3(COOEt)_3$, prepared by boiling 2 grams of the ethyl salt with 10 grams of acetic anhydride, crystallises from ether or alcohol in needles, melts at 75—76°, and is insoluble in cold dilute alkali.

Ethyl triketohexamethylenecarboxylate trioxime,

$$C_6H_3(N\cdot OH)_3(COOEt)_3,$$

is obtained by dissolving the ethyl salt in excess of ammonia, and adding hydroxylamine hydrochloride; the product which is a com-

pound of the oxime with ammonia, is treated with dilute acetic acid. It crystallises in groups of slender, reddish needles, which decompose without melting at 169—171°.

Ethyl bromophloroglucinoldicarboxylate, $C_6Br(OH)_3(COOEt)_2$, is formed as a white precipitate when a solution of bromine in carbon bisulphide is slowly added to a solution of the ethyl tricarboxylate in chloroform. It crystallises from alcohol in slender needles melting at 128°.

When an alkaline solution of ethyl phloroglucinoltricarboxylate is treated with chlorine and the product treated with ammonia, trichloracetamide is obtained (compare Hlasiwetz and Habermann; *Annalen*, **175**, 260). N. H. M.

Action of Ethyl Sodomalonate on Resorcinol. By A. Michael (*J. pr. Chem.* [2], **37**, 469—471).—The author has previously shown that a fluorescent substance is obtained by the action of ethyl sodomalonate on alcoholic resorcinol. This substance is better obtained by mixing equivalent proportions of solid sodium ethoxide and resorcinol dissolved in ethyl acetoacetate, and heating for 10 hours in the water-bath in an atmosphere of hydrogen; the resulting mass is then dissolved in water and acidified, whereby the new compound is precipitated. After several recrystallisations from alcohol, it forms slender, white needles, melting with decomposition at 191—191·5°; it is freely soluble in hot alcohol, sparingly so in cold alcohol and hot water, and insoluble in cold water; its alkaline solution is strongly fluorescent. This substance has acid properties, and is not precipitated by carbonic anhydride from its alkaline solution; its formula is $C_{11}H_8O_5$, and as it splits up, at a temperature a little above its melting point, into methylumbelliferone and carbonic anhydride, it is probably *β-methylumbelliferonecarboxylic acid*; its other properties confirm this supposition.

The formation of this compound by the action of resorcinol on cold alcoholic ethyl sodomalonate is explained by the intermediate formation of acetonetricarboxylic acid, which the author has found to occur when an alcoholic solution of ethyl sodomalonate is allowed to remain in the cold. A. G. B.

Toluidinesulphonic Acids. By J. V. Janovsky (*Ber.*, **21**, 1802—1805).—Paratoluidineorthosulphonic acid, [Me : SO_3H : NH_2 = 1 : 2 : 4], crystallises in triclinic crystals; $a : b : c = 1·2424 : 1 : s$. The metasulphonic acid crystallises in thin needles, which could not be measured; but their optical properties make it probable that they are rhombic.

Orthotoluidinesulphonic acid (Gerner, *Annalen*, **169**, 375) crystallises in large, lustrous, monoclinic prisms; $a : b : c = 0·8889 : 1 : 0·6476$; $\beta = 84°29'$. Paratoluidineorthosulphonic acid gives no colour reaction with ferric chloride in the cold; when warmed, a bluish Bordeaux-red is produced. Other colour reactions of the three acids are given in a table.

When a trace of lead peroxide is added to cold solutions of paratoluidineortho-, paratoluidinemeta-, and orthotoluidine-sulphonic

acids, yellowish-red, wine-red, and rose colours are produced. The last-mentioned acid yields quinone when oxidised, whilst the other acids do not. N. H. M.

Indolecarboxylic Acids. By G. CIAMICIAN and C. ZATTI (*Ber.*, **21**, 1929—1935).—α-Indolecarboxylic acid obtained by Fischer from the hydrazone of pyruvic acid, is prepared by fusing methylketole with 10 to 15 parts of caustic alkali. The product is dissolved in water, nearly neutralised with sulphuric acid, filtered from potassium sulphate, concentrated by evaporation, and precipitated with dilute sulphuric acid. It is crystallised from boiling benzene. The *methyl salt*, $C_9NH_6MeO_2$, crystallises in small needles melting at 151—152°. When the carboxylic acid (3 grams) is boiled with acetic anhydride (15 grams), the compound $C_6H_5{<}\begin{matrix}N-\\CO\end{matrix}{>}$ or $C_6H_5{<}\begin{matrix}N{\cdot}CO\\CO{\cdot}N\end{matrix}{>}C_6H_5$ is formed. This crystallises in almost insoluble, yellow, lustrous needles, which melt at about 312—315°. It is scarcely attacked by boiling with strong aqueous potash, but dissolves readily in hot, alcoholic potash. *β-Indolecarboxylic acid*, $C_9NH_7O_2$, prepared similarly to the α-compound, crystallises in plates, melts at about 214° with evolution of gas, dissolves sparingly in benzene and in boiling water, more readily in ethyl acetate, ether, and alcohol, and is almost insoluble in light petroleum. The *silver salt*, $C_9NH_6AgO_2$, is a white precipitate. The acid is much less stable than the α-acid, and is decomposed by boiling its aqueous solution. It does not yield a picrate, and gives no precipitate with lead acetate.

In the oxidation of scatole with fused potash, indole and α-indole carboxylic acid are formed as well as the β-carboxylic acid.

N. H. M.

Acetyl-compounds of Methylketole and Scatole. By G. MAGNANINI (*Ber.*, **21**, 1936—1939).—Acetylmethylketole (acetyl-α-methylindole), C_8NH_5MeAc [Ac : Me = 1′ : 2′] (Jackson, Abstr., 1881, 734, Fischer, Abstr., 1887, 265), is formed in small amount, together with β-acetyl-α-methylindole, when methylketole is boiled with acetic anhydride and anhydrous sodium acetate; when boiled with aqueous potash (sp. gr. 1·22) it decomposes into methylketole and acetic acid. β-Acetylmethylketole is also formed by the action of acetic chloride on methylindole; in presence of zinc chloride a magenta-like dye is produced, which is possibly related to Fischer and Wagner's rosindole (Abstr., 1887, 588). Acetylscatole [Ac : Me = 2′ : 3′] (this vol., p. 483), dissolves readily in alcohol and acetone, less in ether, and is almost insoluble in cold water; it distils with steam. With hot strong sulphuric acid it gives an intense purple-red solution. The *picrate* crystallises in long, slender, orange-yellow needles. Acetylscatole is not changed by boiling potash; boiling hydrochloric acid decomposes it with formation of free scatole and resin. *Acetylscatoxime*, $C_8NH_6Me{:}N{\cdot}OH$, crystallises from dilute alcohol in small needles melting at 119°. N. H. M.

Conversion of Methylketole into Quinaldine. By G. MAGNANINI (*Ber.*, **21**, 1940—1942; compare Abstr., 1887, 1113).—When

4 grams of bromoquinaldine, from methylketole, is heated with 10 parts of hydriodic acid and some amorphous phosphorus at 180° for 6 to 7 hours, quinaldine is formed, which is purified by means of the picrate. The reaction proves that the bases produced by the action of chloroform and bromoform on methylketole are chloro- and bromo-quinaldines, and that they have the constitution [Me : Cl or Br = 2′ : 3′]. N. H. M.

Methylindolecarboxylic Acids. By G. Ciamician and G. Magnanini (*Ber.*, **21**, 1925—1929).—*α-Methyl-β-indolecarboxylic(methylketolecarboxylic) acid*, $C_8NH_5Me{\cdot}COOH$ [= 2′ : 3′], is prepared by passing a stream of carbonic anhydride through a mixture of 10 grams of methylketole and 3·6 grams of sodium heated at 230° to 240° for 4 to 5 hours; towards the end, the temperature is raised to 300°. The product is treated with alcohol, dissolved in water, steam distilled, acidified with dilute sulphuric acid, and extracted with ether. The carboxylic acid is crystallised from acetone, dissolved in a mixture of benzene and ethyl acetate, and treated with animal charcoal. The colourless solution is then concentrated and precipitated with light petroleum. It is a white, crystalline powder, melts at 170—172° with evolution of carbonic anhydride, dissolves readily in alcohol, ethyl acetate and acetone, sparingly in benzene and still less in water. It is not stable, and decomposes when the aqueous solution is boiled. The *silver salt* is a white, crystalline precipitate. The aqueous solution of the acid shows the following reactions:—With ferric chloride, a brown coloration and precipitate, with copper sulphate an apple-green precipitate, with lead acetate and with mercuric chloride, white precipitates.

β-Methyl-*α*-indolecarboxylic (scatolecarboxylic) acid (Wislicenus and Arnold, this vol., p. 361) is obtained in a manner similar to the above compound from 3 grams of scatole and 1 gram of sodium. It crystallises in needles or plates, melts at 165—167° with evolution of carbonic anhydride, dissolves readily in alcohol and ether, less in benzene, and is almost insoluble in light petroleum. It is more stable than the methylketolecarboxylic acid. When heated with sulphuric acid, a splendid purple-red solution is formed. N. H. M.

3′-Phenylindole. By E. Fischer and T. Schmidt (*Ber.*, **21**, 1811—1812).—*3′-Phenylindole*, $C_{14}H_{11}N$, is obtained when phenylacetaldehydphenylhydrazone is treated with hydrochloric acid (compare this vol., p. 698). It crystallises from hot, light petroleum in white leaves, melts at 88—89°, is readily soluble in alcohol, ether, and benzene, rather sparingly soluble in hot, light petroleum and insoluble in water. It distils under the ordinary pressure with only slight decomposition.

The *picrate* crystallises in dark-red needles melting at 107°. When a pine-chip, treated with an alcoholic solution of the indole, is moistened with cold, concentrated hydrochloric acid, it first turns yellow and after some time deep bluish-violet. 3′-Phenylindole yields an oily nitrosamine when treated with nitrous acid, and when heated with zinc chloride at 170° it is converted quantitatively into the isomeric 2′-phenylindole. F. S. K.

Diphenyl-derivatives. By P. Adam (*Bull. Soc. Chim.*, **49**, 97—102 (compare Abstr., 1887, 589).—*Methyldiphenyl*, C_6H_4MePh, is a colourless, mobile, refractive liquid which boils at 272—277°, and dissolves in ordinary organic solvents. It is very stable towards oxidising agents, but chromic acid converts it into metadiphenylcarboxylic acid, melting at 160—161°. This acid is also formed when the bromine-derivative of methyldiphenyl is boiled with a solution of copper nitrate.

Ethoxymethyldiphenyl, $C_6H_4Ph{\cdot}CH_2{\cdot}OEt$, is prepared by treating methyldiphenyl with bromine and boiling the product with alcoholic potash. It is a colourless, syrupy liquid which is readily soluble in ether and alcohol. The *methyl*-derivative, $C_6H_4Ph{\cdot}CH_2{\cdot}OMe$, is a syrupy liquid with a peculiar smell.

Phenylbenzyl alcohol, $C_6H_4Ph{\cdot}CH_2{\cdot}OH$, obtained by treating the preceding compound with hydrogen iodide, is an odourless, tasteless, thick syrupy liquid which is soluble in chloroform, carbon bisulphide, alcohol, and ether.

Dimethyldiphenyl, $C_{12}H_8Me_2$, is a colourless, mobile liquid which boils at 284—290°, and is more sparingly soluble in methyl alcohol than the monomethyl-derivative. When oxidised with chromic acid it yields an infusible acid which does not sublime. Paradiphenylbenzene melting at 205°, and the isomeric isodiphenylbenzene melting at 85°, were also isolated from the product obtained by the action of aluminium chloride on a mixture of methyl chloride and diphenyl (*loc. cit.*).

Acetyldiphenyl, $C_6H_4Ph{\cdot}COMe$, is formed when ethyldiphenyl (*loc. cit.*) is carefully oxidised with chromic acid; it is converted into metadiphenylcarboxylic acid when further oxidised.

Diethyldiphenyl, $C_{12}H_8Et_2$, is a colourless, mobile liquid which boils at 304—310°. When oxidised with chromic acid, it yields an infusible acid which does not sublime.

Diphenylmethylcarbinol, $C_6H_4Ph{\cdot}CHMe{\cdot}OH$, is obtained by acting on acetyldiphenyl with sodium amalgam in alcoholic solution. It melts at 85—86° and is very readily soluble in alcohol.

Diphenylphenylenecarboxylic acid (*loc. cit.*) yields benzene and the potassium salt of paradiphenylcarboxylic acid when fused with potash.

F. S. K.

New Method for the Preparation of Tetraphenylethylene. By P. de Boissieu (*Bull. Soc. Chim.*, **49**, 681—682).—Tetraphenylethylene can be prepared by brominating diphenylmethane and heating the bromodiphenylmethane which is formed to its boiling point; hydrogen bromide is evolved and, after cooling, the crystalline product is purified by distilling and recrystallising from benzene. The yield is 70 to 80 per cent.

F. S. K.

Hydration of Tolane. By A. Béhal (*Bull. Soc. Chim.*, **49**, 337—339).—Tolane is converted into desoxybenzoïn when warmed with ordinary sulphuric acid at 60°.

F. S. K.

α-Tetrahydronaphthylamine. By E. Bamberger and A. Althausse (*Ber.*, **21**, 1786—1795 and 1892—1904).—*α-Tetrahydronaphthyl-*

amine, $C_{10}H_{11}\cdot NH_2$, is prepared in a manner similar to the β-compound (this vol., p. 599) from 15 grams of α-naphthylamine. The bases, after separation from the hydrochlorides, are distilled in a Glinsky's apparatus. It is a viscous, colourless oil having a slight odour of dimethylaniline, boils at 275° under 712 mm. pressure, dissolves readily in organic solvents, much less in water, and is insoluble in soda. The yield of pure base is 135 grams from 225 grams of commercial α-naphthylamine. It does not react with vegetable dyes. The alcoholic solution reduces silver salts when warmed, gold and platinum salts in the cold. The *hydrochloride* crystallises in tetragonal plates readily soluble in water and alcohol; the *sulphate* with ½ mol. H_2O forms flat needles of a satiny lustre; the *mercurichloride* crystallises in white, lustrous needles readily soluble in hot water; the *oxalate* forms lustrous, white needles which, when heated with water, decompose with formation of an oil having an intense odour of α-naphthylamine; the *picrate*, *nitrate*, and *ferrocyanide* are also described. A carbonate could not be obtained. The *acetyl-derivative*, $C_{10}H_{11}\cdot NHAc$, crystallises in long, matted needles, melts at 158°, and dissolves very readily in the usual organic solvents, rather readily in boiling water.

Phenyl-α-tetrahydronaphthylthiocarbamide, $NHPh\cdot CS\cdot NH\cdot C_{10}H_{11}$, is formed when equimolecular weights of the hydro-base and phenylthiocarbimide are heated for a short time on a water-bath. It crystallises from alcohol in lustrous, thick, concentrically grouped prisms melting at 153°.

Phenyl-α-tetrahydronaphthylcarbamide, $NHPh\cdot CO\cdot NH\cdot C_{10}H_{11}$, prepared from phenyl cyanate, crystallises from benzene in pointed needles melting at 193°; it is readily soluble in benzene and alcohol.

Di-α-tetrahydronaphthylcarbamide, $CS(NH\cdot C_{10}H_{11})_2$, is formed when a mixture of tetrahydronaphthylamine and carbon bisulphide dissolved in alcohol is boiled for some hours; no reaction takes place in the cold. It crystallises in concentrically grouped, lustrous needles, dissolves readily in alcohol and chloroform, less in ether, and melts at 170°.

α-Tetrahydronaphthol, $C_{10}H_{11}\cdot OH$, is prepared by dissolving α-tetrahydronaphthylamine (1 mol.) in a slight excess of very dilute sulphuric acid, and gradually adding sodium nitrite (1 mol.) to the solution carefully kept cool (below 2—3°). The crystals of the sulphate which have separated, redissolve, and the whole acquires a yellowish-red colour. After ½ an hour, sulphuric acid (1 : 1) is added, and the liquid boiled until no more nitrogen is evolved; steam is then passed through it, and the distillate, without being filtered, extracted with ether. The residue from the ether is dissolved in soda and precipitated by carbonic anhydride. It melts at 68·5—69°, boils at 264·5—265° under 705 mm. pressure, and dissolves very readily in the usual organic solvents, sparingly in water. It generally separates from the alcoholic solution as an oil which solidifies to large aggregates of thick, lustrous, silvery, rectangular, monoclinic plates. When exposed to air it becomes superficially rose-coloured.

When the acetyl-derivative of α-tetrahydronaphthylamine is treated with bromine, the compound $C_{12}H_{14}NOBr$ is obtained. It forms

clear, lustrous, strongly refractive prisms, melts at 181°, and dissolves readily in alcohol, rather less readily in benzene. It does not give up bromine when treated with alcoholic potash. α-Tetrahydronaphthylamine is oxidised by potassium permanganate to normal adipic acid, $COOH\cdot[CH_2]_4\cdot COOH$, and oxalic acid. The conversion of α-tetrahydronaphthylamine into naphthylamine is readily effected by means of platinic chloride; oxalic acid seems to produce the same result. The above results make it probable that in α-tetrahydronaphthylamine all four added hydrogen-atoms are present in the benzene-ring to which the amido-group is united. N. H. M.

Acenaphthene and Chlorine. By J. T. Kebler and T. H. Norton (*Amer. Chem. J.*, **10**, 217—218).—Chlorine is rapidly absorbed by acenaphthene, much heat being evolved; the final product is a liquid, very viscous when cold, decomposing on distillation and even on keeping; analyses indicate that it possibly may be trichloracenaphthene. H. B.

Colouring Matter from Anthraquinonedisulphonic Acid and Sodium Nitrite. By J. Walder (*Chem. Zeit.*, **11**, 1530).—This colouring matter is a brownish-red paste containing 25 per cent. of dry matter; it is not soluble in water or acids, but dissolves in alkalis with a reddish-brown, or when concentrated, a black colour. With cotton mordanted with iron or alumina, or both, it gives tints resembling those of alizarin, which are tolerably fast against soap and chlorine. Its tinctorial power, however, is much less than that of alizarin, and on this account it does not seem suitable for the production of full shades. Its composition is still a matter for conjecture. D. A. L.

Terpinol. By Bouchardat and Voiry (*Compt. rend.*, **106**, 1359—1361).—Terpilene dihydrochloride was heated with alcoholic potash at 100° for 24 hours and the product washed with water and distilled under reduced pressure. Two-thirds of the product boils at 175—180°, and one-third at 215—220.°

The first fraction has the composition and properties of citrene. With dry hydrogen chloride at 0°, it yields a dihydrochloride which melts at 47°. This fraction contains no terpane.

The second fraction has a slight odour of terpol, but is more mobile and does not solidify even at a very low temperature; sp. gr. at 0° = 0·924. It is a terpol-derivative of the composition $C_{10}H_{16},C_2H_6O$, and with hydrogen chloride yields ethyl chloride and a dihydrochloride, $C_{10}H_{16},2HCl$.

It is evident that the product of the action of alcoholic potash on terpilene dihydrochloride, although seemingly similar to List's terpinol, is in reality very different. C. H. B.

Essence of Eucalyptus Globulus. By R. Voiry (*Compt. rend.*, **106**, 1419—1421).—The crude essence is a pale-yellow, slightly dextrogyrate liquid with a disagreeable odour; sp. gr. 0·932. It solidifies at −50° to a mass of crystals which melt at about −10°. This latter property is common to all specimens of the essence of

Eucalyptus globulus and also to the essences of many other species. The first fractions contain water, formic and acetic acids, and butyric and valeric anhydrides. The fraction 158—160 consists of terebenthic hydrocarbon with a dextrorotatory power $[\alpha]_D = +40°$; sp. gr. at $0° = 0{\cdot}88$. With hydrogen chloride it yields a crystalline monohydrochloride which melts at 126—128° and has a rotatory power $[\alpha]_D = +27° 30'$. The fraction 170—175° consists of eucalyptol, $C_{10}H_{18}O$, which constitutes about two-thirds of the essence. It is purified by crystallisation at a low temperature and forms a colourless, mobile liquid, which has an odour of menthol or camphor and solidifies at 0° and melts at 41°; sp. gr. = 0·940. It is optically inactive, and with hydrogen chloride yields a hydrochloride, $2C_{10}H_{18}O,HCl$, agreeing in its properties with that obtained by Woelkel from cyneol.

Schimmel's method for the extraction of eucalyptol from the hydrochloride does not give a pure product.

When essence of eucalyptus is distilled under reduced pressure it yields a terpilenol which boils at 130—135° under a pressure of 40 mm., and forms a dihydrochloride, $C_{10}H_{16},2HCl$. The fractions at higher temperatures contain ethereal salts of this terpilenol with acetic, butyric, and valeric acids. The last fractions consist of resins and polymerides of terebenthene. Essence of eucalyptus also contains a sulphur compound which decomposes with evolution of hydrogen sulphide when heated. C. H. B.

Essence of Cajeput. By R. Voiry (*Compt. rend.*, **106**, 1538—1540).—Essence of cajeput is a green liquid with a penetrating odour, a column of 10 cm. having a lævorotatory power of −2°; sp. gr. at $0° = 0{\cdot}934$. It crystallises at −50°, and the crystals melt at −8°.

The fraction at 70—100° contains aldehydes, including butaldehyde and valeraldehyde; the fraction at 155° under ordinary pressure consists of a small quantity of a lævogyrate terebenthene which forms a crystalline monohydrochloride with a rotatory power $[\alpha]_D = -4°$. The fraction at 165° contains benzaldehyde.

The greater part of the essence, however, boils at 175—180°, and consists of *cajeputol* or cajeput terpane, $C_{10}H_{18}O$, identical with the terpane from essence of eucalyptus.

The fraction at 130—140°, under a pressure of 40 mm., has the composition of terpilenol, but will not crystallise, probably owing to the presence of a small quantity of its acetate. If saponified with alcoholic potash at 100° and redistilled, it forms a viscous liquid which boils at 125—130° under a pressure of 40 mm.; sp. gr. at $0° = 0{\cdot}947$. It is an inactive terpilenol which forms a dihydrochloride, and is identical with the terpilenol described by Bouchardat and Lafont.

The higher fractions contain the acetate of this terpilenol together with small quantities of the butyrate and valerate. Hydrocarbons are also present which boil at 160° in a vacuum. C. H. B.

Oil of Peppermint. By A. Jandous (*Chem. Centr.*, 1888, 581, from *Böhm pharm. Zeit.*, **7**, 71).—In commerce a peppermint oil occurs which is without the usual smell, but which is nevertheless

quite pure. The peppermint oil extracted by the author from some specimens of undoubted *Mentha piperita* had a peculiar smell like balm mint, and quite different from the smell of the ordinary peppermint oil.

Mentha forms hybrids with extraordinary ease, and the smell of the oil appears to change also. J. P. L.

Salts of Camphoric Acid. By I. H. Manning and G. W. Edwards (*Amer. Chem. J.*, 233—235).—*Manganese camphorate*, $MnC_{10}H_{14}O_4$, is obtained by heating a mixed solution of manganous sulphate and potassium camphorate; it is less soluble in hot than in cold water, and is not crystalline. *Chromium camphorate*,

$$Cr_2(C_{10}H_{14}O_4)_3,$$

is a heavy precipitate. The basic *ferric salt* is a yellowish precipitate. The *mercuric salt*, $HgC_{10}H_{14}O_4$, is an insoluble, white precipitate. The *aluminium salt*, $Al_2(C_{10}H_{14}O_4)_3$, is formed by boiling aluminium hydroxide with camphoric acid; it is quite insoluble. The *nickel salt*, $Ni(C_{10}H_{14}O)_2$, is prepared by dissolving nickel hydroxide in the acid and heating, when it separates as a crystalline crust. The *strontium salt*, $SrC_{10}H_{14}O_4 + 6H_2O$, is soluble and crystalline.

H. B.

Nitrocamphorates. By P. Cazeneuve (*Bull. Soc. Chim.*, **49**, 92—97; compare Abstr., 1887, 842).—*Sodium nitrocamphorate*, $NO_2{\cdot}C_{10}H_{14}{\cdot}ONa$, prepared by adding sodium carbonate to an alcoholic solution of the zinc salt, crystallises from hot alcohol in large, anhydrous tufts which decompose when heated, are very readily soluble in water and alcohol, but insoluble in ether. Its dextorotatory power is $[\alpha]_D = +289°$. The *potassium* salt, $NO_2{\cdot}C_{10}H_{14}{\cdot}OK$, forms cauliflower-like masses. The *ammonium* salt is obtained in slightly yellowish, amorphous crusts. The zinc salt crystallises in large, white, efflorescent, hexagonal plates containing 1 mol. of water. It is very sparingly soluble in water and ether, soluble in alcohol, and has a dextrorotatory power $[\alpha]_D = +275°$. The calcium, barium, ferrous, ferric, silver, copper, and lead salts are also described.

Quinine nitrocamphorate, $(NO_2{\cdot}C_{10}H_{14}O)_2,C_{20}H_{24}N_2O_2,H_2O$, prepared from the sodium salt and quinine hydrochloride, crystallises in needles which are sparingly soluble in cold water, more readily in hot water, alcohol, and ether. It turns yellow when heated at 127°, and melts at 131° with decomposition. Its dextrorotatory power in a 2·72 per cent. alcoholic solution is $[\alpha]_D = +45·9°$. F. S. K.

Sugars from Hesperidin and Isohesperidin. New Formulæ of Hesperidin and Isohesperidin. By C. Tanret (*Bull. Soc. Chim.*, **49**, 20—23).—Hesperidin and isohesperidin, when boiled with dilute sulphuric acid, yield 50 to 55 per cent. of a sugar and 50 to 55 per cent. of hesperitin, melting at 222° with decomposition The sugar is a mixture of two parts of glucose with one part of isodulcitol. The combined weight of the products was, in every case, greater than that of the glucoside taken, from which it follows that the decomposition takes place with fixation of the elements of

water. From these results the author concludes that the formula of hesperidin is $C_{50}H_{60}O_{27}$, and that of isohesperidin $C_{50}H_{60}O_{27},5H_2O$, both of which agree with the results of elementary analysis.

F. S. K.

Vernonin. By E. Heckel and F. Schlagdenhauffen (*Compt. rend.*, **106**, 1446—1449).—*Vernonia nigritiania* grows on the West Coast of Africa, where it is known as Batiatior or Batjentjor, and is in some repute as a febrifuge. It is a roughly spherical knotted mass of long, greyish-yellow fibres. It contains no emetine and no alkaloïds. If the alcoholic extract is mixed with lime, evaporated to dryness, and extracted with water, the aqueous solution, unlike the original aqueous solution of the root, has no reducing power. If, however, the lime residue is boiled with hydrochloric acid on a water-bath it yields glucose and a resinous substance which is insoluble in water and in dilute acids. The alcoholic extract of the root contains a glucoside which unites with lime to form an insoluble compound.

The glucoside is a slightly hygroscopic, white powder which forms a pale-yellow solution. It is only slightly soluble in ether and chloroform. With sulphuric acid it gives a brown coloration which changes to purple and persists for several hours. The glucoside has the composition $C_{10}H_{24}O_7$, whilst the resinous substance has the composition $C_4H_{10}O_3$, and it follows that when the glucoside undergoes hydrolysis it assimilates 2 mols. H_2O. The resinous compound gives no well-defined colour reactions, except that with sulphuric acid it gives the same coloration as the glucoside.

The glucoside, which the authors name *vernonin*, is the only active principle in the root. When administered by injection it acts like digitalin, strophanthin, and other cardiac poisons, but its activity is only about one-eightieth of that of digitalin. C. H. B.

Action of Chlorine on Pyridine, Piperidine, and their Derivatives. By O. Bally (*Ber.*, **21**, 1772—1777).—Chlorine acts on pyridine dissolved in chloroform, yielding a white, flaky additive compound, $C_5NH_5Cl_2$, analogous to Hofmann's bromine-derivative (Abstr., 1879, 733). When chlorine is passed through an aqueous solution of pyridine methiodide the compound $C_5NH_5MeICl_2$ is formed, identical with that prepared by Ostermayer (Abstr., 1885, 813); it melts at 90° (not 82°). When boiled with water, it is converted into pyridine methochloride, with liberation of iodine. The platinochloride of the latter compound melts at 205—207° (not 188°).

The ammonium iodides of piperidine and quinoline behave similarly. When the chloriodide-derivative, above described, is treated with chlorine in presence of alkali, *pyridine methochloride iodotrichloride*, C_5NH_5MeCl,ICl_3, is obtained; it forms bright yellow, slender needles melting at 179—180°. When dissolved in alcohol, chlorine is eliminated, and the original pyridine methochloride iodochloride (m. p. 90°) is formed.

Chlorine acts on dry piperidine with explosive violence with formation of much soot. When chlorine is passed through piperidine dissolved in water or chloroform, a heavy oil is formed, which sometimes explodes under water, although not dangerously. The new

compound, $C_5NH_{10}Cl$ [Cl = 1], is best prepared by mixing concentrated solutions of bleaching powder and pyridine, extracting the oil with ether, drying quickly with potash, and evaporating off the ether in a vacuum. It has a penetrating, unpleasant odour, and when kept for a few hours deposits white needles of piperidine hydrochloride.

Dichlorohydroxyacetylpiperidine, $OH{\cdot}C_5NH_7Cl_2Ac$ [Ac : OH : Cl_2 = 1 : 2 : 3 : 5], is formed when chlorine is passed through cooled acetyl piperidine (b. p. 224°). An oil, probably trichloracetylpyridine, is formed as intermediate product; when this begins to solidify the whole is allowed to become warm, is afterwards heated on a water-bath, and is finally poured into water. It crystallises from water in colourless rhombohedra, melts at 122°, dissolves readily in alcohol, benzene, ether, acetone, hot water, and in alkalis, and reduces ammoniacal silver solutions. N. H. M.

Condensation of Phenylacetaldehyde with Ammonia and Ethyl Acetoacetate. By A. Jeaurenaud (*Ber.*, **21**, 1783—1784).—*Ethyl benzyllutidinehydrodicarboxylate*, $CH_2Ph{\cdot}C_5NH_2Me_2(COOEt)_2$ [CH_2Ph : Me_2 = 4 : 2 : 6], is obtained by heating 8 grams of phenylacetaldehyde (prepared by Erlenmeyer and Glaser's method (*Annalen*, **147**, 83, 98, and **195**, 140), 17 grams of ethyl acetoacetate, and 20 grams of 8 per cent. alcoholic ammonia in a reflux apparatus on a water-bath under a pressure of 20 cm. of mercury. It crystallises in needles, melts at 115°, and dissolves readily in alcohol, ether, and benzene. When the alcoholic solution is treated with nitrogen trioxide, ethyl lutidinedicarboxylate and benzoic acid are formed. N. H. M.

A New Quinolinequinone. By J. Mathëus (*Ber.*, **21**, 1886—1888).—*Orthonitrosoparahydroxyquinoline*, $NO{\cdot}C_9NH_5{\cdot}OH$ [= 1 : 2], is prepared by gradually adding sodium nitrite (1 mol.) to a cooled solution of parahydroxyquinoline (1 mol.) in dilute hydrochloric acid (2 mols.). After some time, the nitroso-compound is carefully precipitated with ammonia, dried, and crystallised from glacial acetic acid, from which it separates in gold-coloured needles. The yield is almost quantitative. The solutions in soda and in sodium carbonate are green, and deposit the sodium salt in green needles. When reduced with stannous chloride, amidoparahydroxyquinoline is obtained.

Orthonitroparahydroxyquinoline, $NO_2{\cdot}C_9NH_5{\cdot}OH$, is obtained by treating the nitroso-compound with not too dilute nitric acid. It crystallises from the dilute aqueous solution in groups of slender, pale yellow needles, and from more concentrated solutions in reddish-brown, rhombic plates; it melts at 134°, dissolves readily in hot alcohol and dilute acids, sparingly in cold water, and is insoluble in ether and chloroform. The *hydrochloride* crystallises in slender, white needles readily soluble in cold water; the *potassium* and *sodium salts* form splendid orange-coloured needles and yellowish-brown needles respectively.

Quinolinequinone is prepared from parabenzeneazoparahydroxy quinolinesulphonic acid, or from orthamidoparahydroxyquinoline.

The solution of the latter compound in hydrochloric acid is evaporated down a little, allowed to become cold, treated with dilute sulphuric acid, and then with a slight excess of ferric chloride. The reddish-brown crystals which separate are dried on tiles, and treated with hot water and with barium chloride; on evaporating down the filtered solution, the *hydrochloride* separates in long, reddish-yellow needles. The *sulphate* forms yellow, rhombic plates. The compound is decomposed by soda and by sodium carbonate with brown coloration; with ammonia a green solution is obtained, which quickly becomes blue from above. (Compare this vol., p. 851.) N. H. M.

Action of Sulphur on Quinaldine. By W. v. Miller (*Ber.*, **21**, 1827—1828).—When sulphur and quinaldine are heated together hydrogen sulphide is evolved, and a series of compounds formed among which is a basic substance free from sulphur. This compound is probably *diquinaldyl*, $C_{20}H_{16}N_2,H_2O$; it crystallises from hot alcohol in needles, melts at 160—162°, and loses water at 110°. The *picrate* and *platinochloride* of this base crystallise in needles. F. S. K.

Quinoline-derivatives. By H. Weidel and M. Bamberger (*Monatsh.*, **9**, 99—115; compare Abstr., 1887, 847).—When oxygen gas is passed over equal parts of dry quinoline and orthotoluidine hydrochloride, mixed with a small quantity of platinised asbestos, for several hours at 180—190°, the temperature being finally raised to 200—205°, the mass assumes a reddish colour, and contains paramido-metamethyl-α-phenylquinoline (pseudoflavaniline), formed by condensation with elimination of hydrogen as water. Pseudoflavaniline may be readily purified after separation from the unattacked toluidine, by alternately dissolving it in dilute hydrochloric acid and precipitating with ammonia. It is isomeric with flavaniline (Fischer, *Sitz. k. bayer. Akad.*, 1885, 333) and crystallises from hot water, which only slightly dissolves it, in flexible hair-like needles melting at 112° and volatilising above that temperature with partial decomposition. The dihydrochloride, $C_{16}H_{14}N_2,2HCl$, is obtained by dissolving the base in strong, hydrochloric acid; the solution on cooling deposits long, lustrous needles, rapidly decomposed on heating, and when dissolved in hot water forming the monohydrochloride, $C_{16}H_{14}N_2,HCl$, crystallising in small, yellow, monoclinic needles. The platinochloride, $C_{16}H_{14}N,H_2PtCl_6$, forms small, pale orange-coloured needles which dissolve in water without decomposition. The acetyl-derivative, $C_{16}H_{13}N_2Ac$, crystallises in small scales melting at 176—177° (uncorr.).

When treated with nitrous acid and the resulting diazo-compound is decomposed, pseudoflavaniline yields *pseudoflavenol*, $C_{16}H_{12}N{\cdot}OH$, melting at 195—196° (uncorr.). The hydrochloride of this base, $C_{16}H_{13}NO,HCl + 2H_2O$, crystallises from water in pale yellow needles, the platinochloride, $(C_{16}H_{13}NO)_2,H_2PtCl_6$, is a dull yellow, crystalline powder consisting of minute needles, and the acetyl-derivative, $C_{16}H_{12}NOAc$, forms glistening plates melting at 106°. *Nitropseudoflavenol*, $NO_2{\cdot}C_{16}H_{12}NO$, crystallises in small, silky needles melting at 160° (uncorr.). *Hydroxypseudoflavenol* forms colourless scales, in-

soluble in water and dilute acids, and melts at 89°. Pseudoflavenol on oxidation with chromic acid yields 2′-quinolinecarboxylic acid (quinaldic acid, m. p. 156·8°), and on distillation with zinc-dust, a new base of the formula $C_{16}H_{11}N$ is obtained.

Metatoluidine hydrochloride, when heated with quinoline in oxygen gas, behaves like orthotoluidine hydrochloride and forms a condensation product; no such compound, however, could be obtained from paratoluidine. G. T. M.

Phenylquinoline-derivatives. By H. Weidel and G. v. Georgievics (*Monatsh.*, **9**, 138—155; compare Weidel and Bamberger, preceding Abstract).—A gentle stream of oxygen gas is passed over a mixture of parahydroxyquinoline hydrochloride (36 grams), aniline hydrochloride (25 grams), aniline (18 grams), and platinised asbestos (3 grams), at 220° for five or six hours, the temperature finally being raised to 230—235, when no more water and aniline vapours escape; the mixture becomes semi-solid and of a red colour, and contains paramido-α-phenylparahydroxyquinoline, $NH_2{\cdot}C_6H_4{\cdot}C_9NH_5{\cdot}OH$, and amidoparaphenylquinoline, $NH_2{\cdot}C_6H_4{\cdot}C_9NH_6$. In order to separate these substances the product is dissolved in acidified water, the solution is filtered, treated with excess of alkali, and shaken with ether, which dissolves the amidoparaphenylquinoline and any undecomposed aniline. On passing carbonic anhydride through the aqueous solution, the amidophenylhydroxyquinoline is precipitated.

Amidoparaphenylquinoline is insoluble in water, but dissolves readily in ether, benzene, alcohol, acetone, and chloroform, from which it crystallises in forms belonging to the monoclinic system. It melts at 182° (uncorr.), and can be sublimed without decomposition. The hydrochloride, $C_{15}H_{12}N_2,HCl + 2H_2O$, crystallises in bright red needles. When oxidised with permanganate in acid solution, the base yields α-hydroxynicotinic acid, hence the aniline-group is attached to the benzene ring of the quinoline molecule.

Paramido-α-phenylparahydroxyquinoline is best purified by repeated crystallisations of its hydrochloride. The base is only slightly soluble in water, ether, alcohol, benzene, and xylene, but dissolves more readily in amyl alcohol, from which it crystallises in small needles melting with decomposition at about 294°. The hydrochloride, $C_{15}H_{12}N_2O,HCl + \frac{1}{2}H_2O$, crystallising in dark red granules, and the sulphate, $(C_{15}H_{13}N_2O)_2,H_2SO_4 + 1\frac{1}{2}H_2O$, in red needles, are both powerful dyes; the acetyl-derivative, $C_{15}H_{10}NOAc_2$, crystallises from alcohol in almost colourless scales. On treating a solution of the base in hydrochloric acid with potassium nitrite and decomposing the resulting diazo-compound, *parahydroxy-α-phenylparahydroxyquinoline* is formed. It crystallises from alcohol in slender, yellowish-white needles, scarcely soluble in water, and melting at 247° (uncorr.).

The constitution of paramido-α-phenylparahydroxyquinoline is shown by the reduction of the corresponding hydroxy-compound, $(C_{15}H_{11}NO_2)$, with zinc-dust, whereby α-phenylquinoline is formed, and further by the production of parahydroxybenzoic acid, when the hydro-compound, resulting from the action of tin and hydrochloric acid on the hydroxy-compound, is fused with potash. G. T. M.

Naphthaquinolines. By A. Combes (*Compt. rend.*, **106**, 1536—1538).—*Dimethyl-α-naphthaquinoline.*—α-Naphthylamine and acetylacetone in molecular proportions are heated on a water-bath for a short time, and the viscous liquid thus obtained is heated at 100° for half an hour with excess of concentrated sulphuric acid. The product is poured into a large excess of cold water, in which it dissolves completely, and if this solution is made slightly alkaline with ammonia, it deposits long, white crystals, which when dried melt at 44° and boil without decomposition at 360—362°. Dimethyl-α-naphthaquinoline forms well-crystallised salts, the solutions of which show a violet fluorescence. The hydrated platinochloride is a bulky, violet substance, which loses water in a vacuum, and forms a violet-brown, amorphous powder which melts at 260° with decomposition.

Dimethyl-β-naphthaquinoline is obtained in a similar manner from β-naphthylamine. It melts at 66—67°, and boils at 380°, becoming red, but can be obtained colourless by distillation in a vacuum. The platinochloride is greenish-yellow, and decomposes without melting at 220°.

In the preparation of dimethylnaphthaquinoline a golden-yellow product is obtained, insoluble in dilute acids, but soluble in alkalis, forming a colourless solution. Acids precipitate yellow crystals, which are also obtained from the ammoniacal solution by simply boiling it. This compound is the sulphonic acid of the naphthaquinoline, $C_{15}H_{13}SO_3$, and can be obtained directly by the action of the acid on the base. The exact constitution of dimethyl-β-naphthaquinoline was not determined.

Acetylacetone may be replaced in these reactions by any of its mono-substitution derivatives. C. H. B.

Action of Silicon Tetrafluoride on Quinine dissolved in Different Liquids. By A. Cavazzi (*Chem. Centr.*, 1888, 664—665, from *Annal. Chim. Farm.*, **6**, 341—346).—Silicon tetrafluoride, when led over dry quinine, has no action whatever. Carbon bisulphide was chosen as a solvent, because Knop and Wolf found that silicon tetrafluoride acted on absolute alcohol, forming tetraethyl silicate and hydrofluosilicic acid, and the author found that it acted on ether after long contact, forming hydrofluosilicic acid and a soluble ethereal silicate.

1 gram of quinine was dissolved in 60 c.c. of carbon bisulphide, and silicon tetrafluoride led over the solution at a distance of 2 cm. from the surface of the solution, which was frequently agitated. The gelatinous precipitate which contained the whole of the quinine was filtered, washed, and dried. It formed a white, amorphous substance, insoluble in carbon bisulphide and ether, but easily soluble in water, which decomposed it with the formation of silicic acid and quinine silicofluoride. This substance melts at a moderately high temperature, and decomposes at higher temperatures with disengagement of silicon tetrafluoride and development of a beautiful carmine-red vapour. Concentrated sulphuric acid decomposes it, with the formation of quinine sulphate and silicon tetrafluoride. In moist air it slowly

becomes sticky, and forms quinine silicofluoride. The author regards it as a mixture of the two compounds $C_{20}H_{24}N_2O_2,SiF_4$ and

$$C_{20}H_{24}N_2O_2,2SiF_4.$$

If equimolecular weights of quinine and hydrofluosilicic acid are evaporated to dryness at 100°, the normal quinine silicofluoride, $C_{20}H_{24}N_2O_2,H_2SiF_6$, is formed. A better process for obtaining this salt is the following:—Pass silicon tetrafluoride over a solution of quinine in 50 c.c. of absolute alcohol, until the flocculent precipitate which first forms is redissolved, then heat the solution at 40°, and shake, the salt is immediately precipitated as a white, coarse-grained powder. If instead of 50 c.c. of absolute alcohol, 100 c.c. be used, and the solution is not agitated, the normal salt soon separates out in glittering crystals. It is insoluble in ether and carbon bisulphide, soluble in cold absolute alcohol, more readily soluble in boiling alcohol, easily in water. Dilute solutions are strongly fluorescent, and leave on evaporation the unchanged salt. It is hygroscopic, and behaves like the first-mentioned substance both on heating and treatment with sulphuric acid. A watery solution precipitates barium from solutions of its salts, a reaction which might prove useful in the determination of that metal under certain circumstances. The neutral quinine silicofluoride can also be prepared by passing silicon tetrafluoride over an ethereal solution of quinine. J. P. L.

Cinchonibine. By E. Jungfleisch and E. Léger (*Compt. rend.*, **106**, 1410—1411; compare this vol., 380, 507, 729).—*Cinchonibine*, $C_{19}H_{22}N_2O$, is isomeric with cinchonine; it is obtained by dissolving the recrystallised succinate in dilute hydrochloric acid, and precipitating with sodium hydroxide. It forms small, colourless, highly refractive, anhydrous, rhomboidal prisms, which melt at about 259°, and if heated rapidly, sublime without sensible decomposition. It is alkaline to litmus, but not to phenolphthaleïn, and is insoluble in water, dilute alcohol, ether, acetone, or chloroform, but dissolves in hot concentrated alcohol. The rotatory power of a 0·75 per cent. solution in alcohol of 97° at 18° is +175°·93; of a 0·5 per cent. solution +176°·06. A 1 per cent. aqueous solution, containing two equivalents of hydrochloric acid, has a rotatory power +220°·53; with four equivalents of acid +224°·84.

The basic salts are slightly alkaline to litmus; the normal salts are acid. The *basic hydrochloride* and *hydrobromide* crystallise in silky needles, very soluble in water; the *platinochloride*, $(C_{19}H_{22}N_2O,PtCl_4)_2 + 3H_2O$, forms small yellow crystals; the *aurochloride* is pale yellow, and only slightly soluble; the *basic thiocyanate*, $C_{19}H_{22}N_2O,CNSH$, forms long, flattened, anhydrous prisms which melt at 203°; the *basic succinate*, $C_{19}H_{22}N_2O,C_4H_6O_4 + 6H_2O$, is only slightly soluble in cold water, especially in presence of an alkaline succinate, but dissolves readily in water containing succinic acid or in alcohol, and crystallises from either solution in large, hexagonal crystals, or from a boiling aqueous solution in prismatic needles, which melt at 207° when dry; the *basic tartrate* crystallises with 1 mol. H_2O in silky needles, which melt at 214° if dry, and are soluble in water; the *basic oxalate* forms

long, brilliant needles, somewhat soluble in cold water; the *basic chromate* is oily, and only slightly soluble; the *malate* crystallises distinctly; the *phosphate* and *arsenate* are only slightly soluble; the *molybdate*, *picrate*, *benzoate*, and *salicylate* are insoluble and amorphous.

Cinchonibine methiodide, $C_{19}H_{22}N_2O,MeI$, is obtained by the direct union of its constituents at the ordinary temperature, or more readily on heating. It crystallises from boiling water in colourless, anhydrous needles, only slightly soluble in cold, but very soluble in hot water, and soluble in methyl or ethyl alcohol. It melts at 252°. The *dimethiodide* is obtained by using a large excess of methyl iodide, and forms pale-yellow lamellæ, which become red when heated but regain their original colour when cold. They melt at 223°, and are very hygroscopic. The *ethiodide* is obtained in a similar manner, and forms long, colourless, anhydrous needles, which melt with decomposition at 245°, and are slightly soluble in cold water. Crystals which separate from a hot solution are anhydrous, but if formed in a cold solution they contain 1 mol. H_2O. The *diethiodide* crystallises in anhydrous, yellow needles, which melt at 251°. The corresponding bromine compound is obtained by heating its constituents together in presence of alcohol. It forms colourless, anhydrous, crystalline crusts, which melt at 215°, and are very soluble in water, but only slightly soluble in ethyl alcohol. C. H. B.

Piperidine. By E. Lellman and W. Geller (*Ber.*, **21**, 1921—1923).—When piperidine (5 grams) is heated with nitrobenzene (22 grams) at 250—260° for four hours it is converted into pyridine.

When piperidine diluted with an equal volume of water is added by drops to a boiling alkaline solution of bleaching powder, and the product steam distilled, the compound $C_5NH_{10}Cl$ (termed by the author piperylene nitrogen chloride) is obtained. This boils at 52° under 25 mm. pressure with slight decomposition, and dissolves readily in alcohol, benzene, ether, and glacial acetic acid. When the solution in much benzene is treated with aluminium chloride, a very small amount of an oily substance, probably tertiary phenylpiperidine, is formed. N. H. M.

Piperylene Nitrogen Chloride. By E. Lellman (*Ber.*, **21**, 1924—1925).—Piperylene nitrogen chloride (preceding Abstract) is decomposed by water with formation of much piperidine and a crystalline base, boiling at 150°. When heated with strong hydrochloric acid, chlorine is evolved, and a chloro-derivative is formed, which soon decomposes, and white needles of piperidine hydrochloride gradually separate. N. H. M.

Conversion of Hyoscyamine into Atropine. By E. Schmidt (*Ber.*, **21**, 1829).—The author succeeded some time ago in converting hyoscyamine into atropine by heating it for six hours at a temperature above its melting point (compare Will, this vol., p. 855). F. S. K.

Ergotinine and Cornutine. By E. Bombelon (*Chem. Centr.*, 1888, 472, from *Pharm. Zeit.*, **33**, 109).—Both alkaloïds can be

obtained by treating powdered ergot free from fat with alcohol of 95°, in which 50 grams of soda is dissolved, and allowing the mixture to remain for 24 hours. The extract must be again treated with cold alcohol without soda; it contains sphacelinic acid, both alkaloïds, some fat, resin, and extractives; it is then acidified with citric acid and distilled to remove the alcohol. The watery solution remaining is filtered (the residue contains the sphacelinic acid), and the filtrate supersaturated with soda and shaken with ether. Ergotinine with some cornutine is dissolved. To separate these alkaloïds completely, the dried ergotinine must be again taken up with absolute ether. The alkaloïds are only stable when combined with acids; weak watery solutions of the alkaloïds quickly decompose. J. P. L.

Hæmatoporphyrin. By M. Nencki and N. Sieber (*Monatsh.*, **9**, 115—132).—The authors have previously (Abstr., 1883, 69) ascribed the name hæmatoporphyrin to the substance of the formula $C_{32}H_{34}N_4O_5$, obtained by the action of concentrated sulphuric acid on hæmatin, $C_{32}H_{32}FeN_4O_4$, or hæmin, $C_{32}H_{30}N_4FeO_3$. They now find that when hæmin (or hæmatin, which, however, does not give so good a yield) is treated with a saturated solution of hydrogen bromide in glacial acetic acid, it is readily converted into true hæmatoporphyrin, which shows properties quite different from those previously described. It has the formula $C_{16}H_{18}N_2O_3$, is insoluble in water and dilute acetic acid, is only slightly soluble in ether, amyl alcohol, and chloroform, but is easily dissolved by alcohol, dilute mineral acids, and solutions of the alkalis and alkaline carbonates. The compound is of a reddish colour, is amorphous and very unstable, turning brown and refusing to dissolve in alkalis and hydrochloric acid, after having been heated at 100°. The solution in alcohol or alkalis shows the four absorption-bands described by Hoppe-Seyler and Nobel. The hydrochloride, $C_{16}H_{18}N_2O_3,HCl$, crystallises in tufts of needles, the sodium salt, $C_{16}H_{17}NaN_2O_3 + H_2O$, in microscopic prisms. The authors have not yet determined whether the compound formed by the action of sulphuric acid on hæmin and hæmatin (*loc. cit.*) is a mixture or is an anhydride of hæmatoporphyrin ($C_{32}H_{34}N_4O_5 = 2C_{16}H_{18}N_2O_3 - H_2O$).

Hæmatoporphyrin has the same empirical formula as bilirubin, which it resembles in many of its properties. When introduced into the living subject, hæmatoporphyrin is partly expelled in the urine, but the greater portion is retained and is probably utilised in the formation of hæmoglobin. G. T. M.

Synthetical Studies on Albuminoïds and Proteïds. By P. Schutzenberger (*Compt. rend.*, **106**, 1407—1410).—The author's investigations of the desintegration of albuminoïds when boiled with barium hydroxide, show that the products are (1), the products of hydration of urea and oxamide; (2), leucines, and (3), leuceïnes, the two latter being present in equivalent proportions. In order to effect the synthesis of albuminoïds it will be necessary to combine a molecule of a leucine with a molecule of a luceïne, with elimination of water, and to then combine this complex group with one or more molecules of urea or oxamide, also with elimination of water.

In order to determine the constitution of the leuceïnes, the zinc salts of the lower leucines, such as glycocine and alanine, were heated at 140—160° in sealed tubes for six hours with zinc oxide and ethylene bromide. The excess of ethylene bromide was poured off, the product dissolved in water and filtered, zinc removed by hydrogen sulphide, barium by sulphuric acid, and bromine by silver carbonate, and the solution was evaporated to dryness in a vacuum. The residue was extracted with alcohol, and the product purified by repeated evaporation and treatment with alcohol. The product is a deliquescent, semi-solid substance, very soluble in alcohol, with all the properties which characterise the leuceïnes obtained from albumin or gelatin. Its solution becomes brown if evaporated in contact with the air. If heated at a high temperature in a current of hydrogen it yields only hydropyrrolic bases, similar to those obtained from leuceïnes. The reactions which take place in the formation of this product may be represented by the equations $NH_2{\cdot}CH_2{\cdot}COOH + C_2H_4Br_2 = 2HBr + C_4H_7NO_2$; $C_3H_7NO_2 + C_2H_4Br = 2HBr_2 + C_5H_9NO_2$; $2NH_2{\cdot}CH_2{\cdot}COOH + 2C_2H_4Br_2 = 4HBr + C_8H_{14}N_2O_4$.

C H. B.

Chemical Nature of the Peptones and the Separation of Albumin from them. By R. Palm (*Zeit. anal. Chem.*, **27**, 359—363).—According to the author's view peptone is a solution of proteïn in an acid. It can be obtained by the action of lactic acid on the albumin of eggs, milk, or blood, also on gelatin, fibrin, or chondrin. An alcoholic solution of peptone mixed with enough ether gives an oily precipitate containing proteïn and lactic acid in constant proportions. A peptone solution, neutralised with ammonia, is coagulable by heat, and gives all the reactions of an albumin solution. Mixed with strong alcohol all the albumin is precipitated. This accounts for the fact that peptones show, on analysis, the same composition as the proteïds from which they are obtained. Solutions of peptones reduce Fehling's solution. Since cow's milk sometimes contains as much as 1·5 per cent. of peptone, determination of the sugar by that solution gives too high a result.

Peptone solutions are precipitated by potassium xanthate; albumin gives no precipitate until after the addition of an acid.

M. J. S.

Physiological Chemistry.

Acids in Healthy and Disordered Stomachs during a Carbohydrate Diet. By T. Rosenheim (*Chem. Centr.*, 1888, 527—528, from *Arch. path. Anat.*, **111**, 414—433).—Ewald and Boas found the following relations in the acids in the gastric juice of a healthy person when a diet of 60 grams of roll and water was taken on an empty stomach. Lactic acid develops very early, and increases for a period of 30 to 40 minutes, during which time hydrochloric acid is

absent, there is then an intermediate period when both acids are present, and a final phase, which lasts to the complete elimination of the stomach contents, when hydrochloric acid only is present. A certain antagonism seems to exist between lactic and hydrochloric acids, which leads to the disappearance of the lactic acid. In opposition, however, to this result, the acidity of the stomach was found to be due to hydrochloric acid alone, which could be detected after 10 minutes, when a 1 to 2 per cent. boiled starch solution was taken.

According to the author's experiments with amylaceous diets, hydrochloric acid appears at a very early stage, it soon reaches a moderately large quantity (above 1 in 1000), and remains essentially the same until the complete passage of the food into the duodenum. Lactic acid is present during all stages of the digestion; the amount which remains fairly constant may, towards the end of the digestion, increase, but it never reaches 1 part in 1000; in every stage of the digestion it is less than the amount of hydrochloric acid.

In pathological conditions, too, the hydrochloric acid can be detected in the very early stages. The amount of hydrochloric acid, both under normal and pathological conditions, is sensibly less when a diet of 25 grams of wheaten starch and 5 grams of sugar or starch only is substituted for the 50 grams of white bread given in the above experiments. In opposition to Ewald and Boas, it is found that the presence of lactic acid in every stage of the digestion, in more or less important quantity, during a purely starch diet, is independent of the amount of hydrochloric acid. J. P. L.

The Fat Equivalent of Starch (Fattening of Sheep). By T. Pfeiffer and F. Lehmann (*Bied. Centr.*, 1888, 374—383).—The general results of the experiment made to estimate the fat equivalent of starch, show that Henneberg's factor 2·5 is accurate, and that the nutritive values of saccharose and the other carbohydrates in roots, bran, &c., are to all intents and purposes equal. A table shows the increase of weight of the various parts of the animal as influenced by feeding with sugar and with fat. Sugar appears to increase the consumption of water. E. W. P.

Formation and Change of Alcohol and Aldehyde in the Organism. By Albertoni (*Chem. Centr.*, 1888, 529, from *J. Acad. Belg.*, 1887).—On distillation of fresh or putrid human or animal intestine, a substance was obtained which gave the iodoform reaction, occasionally very markedly, but which could not be an alcohol, as it did not give Vitali's reaction.

Alcohol could only be detected in the breath, urine, and other parts of the body when large doses were given to the animals experimented on; it almost entirely disappears in the organism. Aldehyde, on the other hand, can always be detected in the breath and urine, even when only small quantities have been absorbed, either by the stomach or skin. It is completely excreted by the lungs and kidneys unchanged. If alcohol underwent, according to Liebig's theory, the successive changes into aldehyde, acetic acid, and carbonic anhydride, aldehyde would certainly be found in the breath and

kidneys after alcohol had been taken, which is never the case. The formation of alcohol, either in living or putrefying tissue, is a very rare circumstance. The product mentioned above, which gives the iodoform reaction, is neither alcohol, aldehyde, nor acetone.

J. P. L.

Occurrence of Lactic Acid in Blood and its Formation in the Organism. By M. Berlinerblau (*Chem. Centr.*, 1888, 757—758, from *Centr. med. Wiss.*, 1888, 317).—The author has found lactic and sarcolatic acids to be constant constituents of blood: rabbit's blood contained 0·065 to 0·072 per cent., dog's blood, 0·71 per cent., and human venous blood, 0·008 per cent.

Artificial circulation experiments with blood, to which glucose and glycogen had been respectively added, showed that after three hours' circulation a small amount of lactic acid was formed in the case of glucose, and sensibly more with glycogen. Under similar conditions sodium propionate and butyrate were without influence, the greater part of both substances being found afterwards in the blood unchanged.

J. P. L.

Coagulation of the Blood. W. D. Halliburton (*Proc. Roy. Soc.*, **44**, 255—268).—The present research was directed to the determination of the nature of the ferment that produces the change of fibrinogen into fibrin. It was found that lymph cells contained the following proteïds:—(1.) A mucin-like proteïd, similar to that described by Miescher (*Med. Chem. Unters.*, 441). Further experiments showed that this proteïd belongs to the class of nucleo-albumins recently described by Hammarsten (this vol., pp. 167, 170). (2.) Two globulins; one which occurs in small quantities enters into the condition of a heat coagulum at about 50°, and the other at 75°. To the latter the name *cell-globulin* is given. (3.) An albumin, to which the provisional name of *cell-albumin* is given. No substance like myosin or fibrin can be obtained from the cells; there is, however, a formation of sarcolactic acid after death, as in muscle; and if the glands are left, especially at the temperature of the body, for some hours after death, a process of self-digestion takes place, the pepsin present in the glands, as it is in most tissues (Brücke), becoming active when the reaction of the tissue becomes acid; under these circumstances there is in addition to the proteïds already enumerated a small and varying amount of albumoses and peptones.

In order to ascertain what action, if any, was exerted on the coagulation of the blood by the various proteïds that occur in the cells of lymphatic glands, the following experiments were made:—A watery or saline extract of fresh glands had very considerable clotting powers; that is to say, the addition of a few drops of such an extract caused diluted salted plasma to clot in a few minutes, which otherwise did not clot until after the lapse of 12 to 24 hours. The activity of this extract was not altered by heating to 70°; it was therefore independent of the nucleo-albumin which is disintegrated at about 50°, or of the globulin which coagulates at that temperature. Its activity was destroyed, however, if heated above 75°. Extracts of both dried and fresh glands contain a substance which has the

same properties as fibrin ferment, and which, moreover, is rendered inactive at the temperature at which fibrin ferment, as ordinarily prepared from serum, loses its activity.

The next question investigated was whether the ferment action was dependent on, or independent of, the presence of the proteïds of the cells. An extract of the cells was made with sodium sulphate solution, and saturated with ammonium sulphate; the precipitate of the proteïds so produced was filtered off; the proteïd-free filtrate dialysed till free from excess of salt, and it was then found to have no power of hastening coagulation. The precipitate, which contained all the proteïds, was washed with saturated solution of ammonium sulphate, and redissolved by adding distilled water; this solution hastened the coagulation of salted plasma very considerably. This experiment showed either that the ferment was identical with or precipitated with the proteïds in the extract. It was, moreover, destroyed at a temperature at which these proteïds were coagulated, namely, about 75°; there are, however, in the solution, two proteïds which are coagulated at about this temperature, the cell-globulin and the cell-albumin. These were separated by the magnesium sulphate method, and it was found that the globulin and not the albumin has the properties of a fibrin ferment. The question then naturally arose whether this cell-globulin is the same as the substance which has been termed fibrin ferment prepared from serum. A number of experiments were performed with fibrin-ferment prepared in various ways, and the final conclusions drawn are as follows:—

1. Lymph cells yield as one of their disintegration products a globulin which may be called cell-globulin. This has the properties that have hitherto been ascribed to fibrin ferment.

2. Fibrin ferment as extracted from the dried alcoholic precipitate of blood serum is found on concentration to be a globulin with the properties of cell globulin.

3. The fibrin ferment as extracted by saline solutions from "washed blood clot" is a globulin which is also identical with cell globulin.

4. Serum globulin as prepared from hydrocele fluid has no fibrinoplastic properties. It may perhaps better be termed plasma globulin.

5. Serum globulin as prepared from serum has marked fibrinoplastic properties. This is because it consists of plasma globulin, and cell globulin derived from the disintegration of white blood corpuscles, which are in origin lymph cells.

6. The cause of the coagulation of the blood is primarily the disintegration of the white blood corpuscles; they liberate cell globulin which acts as a ferment converting fibrinogen into fibrin. It does not seem to become a constituent part of the fibrin formed.

With regard to Wooldridge's theory of the coagulation of the blood (this vol., p. 305), it is pointed out that lecithin causes a hastening of no other form of plasma but peptone plasma, and it is held that this form of plasma is a very objectionable one to use, both on account of the presence of numerous proteïds foreign to plasma (albumoses and peptones), and on account of alterations in the normal proteïds, as is seen by their heat coagulation temperatures. It is shown that the

elaborate hypothesis of the transference of lecithin from one so-called fibrinogen to another in order to form fibrin, is a pure assumption, and supported by no analytical evidence. The occurrence of a precipitate on cooling plasma (Wooldridge's fibrinogen A) is also considered to be the result of the hetero-albumose present in the commercial peptone used. The mere cooling of a solution of Witte's peptone causes such a precipitate, and this, moreover, consists of rounded granules similar in appearance to blood-tablets. It is urged that the word fibrinogen should be restricted to the substance described by Schmidt and Hammarsten under that name, and that the group of substances obtained by Wooldridge from the tissues (this vol., p. 618), are probably nucleo-albumins, which when injected into the circulation form strings of a mucinoid description in the vessels, which form the starting point for the thrombosis or intravascular coagulation which occurs. W. D. H.

Malic Acid in Suint. By A. Buisine and P. Buisine (*Compt. rend.*, **106**, 1426—1428).—Suint contains malic acid in the form of a potassium salt to the extent of 2·5 per cent. of the solid residue. It also contains succinic acid in similar quantity. The potassium malate is a product of secretion, and is not produced by the fermentation of other constituents of the suint. It is most probable that the malic acid is derived from the food of the sheep, and passes unaltered through the system until it is excreted in the perspiration. C. H. B.

Animal Melanin. By M. Nencki and N. Sieber (*Chem. Centr.*, 1888, 587, from *Arch. expt. Path. Pharm.*, **24**, 17).—Hypomelanin gives *hypomelaninic acid* when melted with potash. The acid is soluble in ammonia, reprecipitated by hydrochloric acid, and contains more carbon and less sulphur than melanin. On purifying the acid, the carbon increases, the sulphur decreases. An attempt to isolate as a chemical product the black pigment of horse's hair failed. An amorphous acid, *sepiaic acid*, was obtained by digesting for a long time the colouring matter from the ink bag of sepia with 15 times its weight of 10 per cent. potash. This acid is soluble in alkaline solutions, and is precipitated from ammoniacal solutions by ammoniacal zinc chloride or copper sulphate. It contains 56·3 per cent. C, 3·6 per cent. H, 12·33 per cent. N, 0·51 per cent. S, and 27·2 per cent. O. The melanin obtained from a melanotic sarcoma of the liver was not identical with phymatorhusin found in previous cases; this is also true of the melanin obtained from sarcomatous inguinal glands and the black pigment deposited in the skin in Addison's disease. J. P. L.

Buffalos' Milk and Butter. By F. Strohmer (*Chem. Centr.*, 1888, 478, from *Zeit. Nahr. Hygiene*, **2**, 17—19).—In Hungary and Transylvania buffalo's milk is commonly used. A sample analysed by the author gave the amphioteric reaction, had a pleasant taste, but a musk-like smell. Microscopically no difference could be observed between the fat corpuscles of buffalo's milk and ordinary cow's milk; the specific gravity of the milk at 15° was 1·0319, and it contained 81·67 per cent. water and 18·33 per cent. of solid residue; the solids

contained 9·02 per cent. fat, 3·99 per cent. caseïn, 4·50 per cent. milk-sugar, and 0·77 per cent. ash. The nitrogen was 0·6 per cent., phosphoric acid 0·3 per cent. Another sample, which had become sour before analysis, gave 18·01 per cent. total solids, 8·54 per cent. fat, and 0·62 per cent. nitrogen. The two points in which buffalo's milk, therefore, differs from cow's milk are the high percentage of fat and the musk-like smell.

Butter and cheese made from the milk do not keep well. The fat of buffalos' milk when treated according to Meissl's process was found to contain volatile fatty acids equivalent to 33·6 c.c. of decinormal soda. A salt butter made in Transylvania gave on analysis 17·67 per cent. water, 80·98 per cent. fat, 1·19 per cent. caseïn and milk-sugar, and 0·16 per cent. ash. The acidity of 100 grams of butter was equal to 23·43 c.c. of normal potash. The butter-fat melts at 31·3°, and congeals at 19·8°. The fatty acids melt at 37·9°. Köttstorfer's equivalent = 222·4 mgrms. of potash. Richert-Meissl's equivalent = 30·4 c.c. of decinormal alkali. The fat does not essentially differ from the fat of ordinary cow's milk either in its chemical or physical properties. J. P. L.

Excretion of Iron from the Animal Organism and the Amount of this Metal in starving Animals. By S. Zaleski (*Chem. Centr.*, 1888, 759, from *Arch. Exp. Path. Pharm.*, **23**, 317).—3 c.c. of a solution of sodiotartrate of iron containing 0·0096 gram of iron, was injected into the jugular vein of one of two rabbits of equal weight, fed on the same diet and allowed to fast four days prior to the experiment. Both animals were killed three hours afterwards and the whole circulatory system freed from blood. An analysis of the various organs of both animals showed that in the case of the rabbit which had received the injection of iron the liver was the only organ that contained more iron than the corresponding organs of the control animal. The author draws the conclusion that iron, in contradistinction to other heavy metals, is excreted by the liver and not by the intestine.

J. P. L.

Secretion of Perspiration by the Skin after taking Alcohol. By G. Bodländer (*Chem. Centr.*, 1888, 759—760, from *Zeit. Klin. Med.*, **13**, Heft 5).—An apparatus for readily determining the amount of perspiration from any part of the skin is described. By means of this apparatus the author has found that in the majority of cases alcohol causes a diminution of the amount of perspiration. No ratio, however, between the amount of alcohol taken and the perspiration given off has been observed.

On account of this power of diminishing, in the majority of cases, the amount of perspiration, alcohol may be useful in the case of profuse perspiration of debilitated subjects. J. P. L.

Toxic Action of Chlorates. By F. Marchand (*Chem. Centr.*, 1888, 472—473, from *Arch. Exp. Path. Pharm.*, **22**, 201—232, and **23**, 273—316).—Stockvis has stated that chlorates have no specially toxic properties, sodium chlorate acting like sodium chloride and

potassium chlorate showing only the toxic action of all potash salts; methæmoglobin did not arise in the living blood but only in the dead blood after removal from a vein.

The author points out that Stockvis only experimented on rabbits, the strong alkalinity of whose blood perhaps accounted for the slight action.

When the blood of poisoned animals (dogs) was tested, the methæmoglobin bands were immediately recognisable, so that the author concludes that the living blood undergoes a pathological change. The more rapidly the chlorate is absorbed into the blood, the quicker its action. A quantity of chlorate which when injected into a vein in the course of 73 minutes produced scarcely any result, produced strong symptoms of poisoning when the period of injection was reduced to 30 minutes, and a fatal result when the time was further reduced to 10 minutes.

A similar quantity of chlorate when given by the stomach soon produced changes in the blood and secretion of blood pigment in the urine. The changes in the blood are effected most rapidly by intraperitoneal injection.

Rabbits and guinea-pigs on account of their sensitiveness to irritation of the digestive organs may succumb to gastritis induced by the corrosive action of the chlorate before absorption has taken place.

J. P. L.

Action of Chlorates. By J. Cahn (*Chem. Centr.*, 1888, 473, from *Arch. Exp. Path. Pharm.*, **24**, Heft 3).—Chlorates did not cause the appearance of any methæmoglobin in the blood or urine of rabbits even when hydrochloric acid had been administered to reduce the alkalinity of the blood.

The author corroborates Marchand's results (preceding Abstract), namely, that methæmoglobin very readily occurs when chlorates are administered to dogs.

During marked diuresis more than 3·5 per cent. of sugar was found in the urine. The probable explanation of this is that the chlorate produces changes in the blood similar to those produced in poisoning with amyl nitrite.

J. P. L.

Chemistry of Vegetable Physiology and Agriculture.

Antiseptic Properties of the Naphthols. By J. Maximovitch (*Compt. rend.*, **106**, 1441—1443).—Numerous experiments show that α-naphthol in the proportion of 2·5 grams and β-naphthol in the proportion of 5·0 grams per litre will kill the microbes in ordinary cultivation liquids and jellies. Their efficiency is greatest in liquids. Different microbes vary considerably in their power of resisting the antiseptic. The microbe which produces a green coloration offers the greatest resistance, and is only killed by 2·5 grams of α-naphthol, or 3·5 grams of β-naphthol per litre. Other patho-

genic microbes require 0·2 to 0·7 gram of α-naphthol and 0·8 to 1·5 gram of β-naphthol per litre. C. H. B.

Origin and Fate of Nitric Acid in Plants. By B. Frank (*Chem. Centr.*, 1888, 336, from *Ber. deut. bot. Ges.*, **5**, 472—487).—Contrary to the general view taken as to the origin of nitric acid in plants, experiments conducted by the author tend to show that nitrates are never formed in the plant, but that the nitric acid must be presented to the plant ready formed. It is then assimilated by the roots, from which it passes to the upper parts by means of the larger cell systems of the stem and leaf ribs. It is these organs which collect the excess of nitrate above that which is required for the healthy growth of the plant. The author points out that in plants such as the ash, in which nitrates are absent from the upper parts, they may yet be detected in the finer rootlets. J. W. L.

Quantitative Estimation of the Heat and Carbonic Anhydride given out by Parts of Plants. By H. Rodewald (*Bied. Centr.*, 1888, 384—385).—From comparisons of the amount of heat evolved with the carbonic anhydride exhaled by apples, the author comes to the conclusion that the energy set free by the exhalation of gas is in great part converted into heat and external work; this latter may either appear as evaporation of water, or in some cases may be partially utilised in overcoming mechanical obstacles to free growth. E. W. P.

Slow Combustion of Organic Substances. By T. Schloesing (*Compt. rend.*, **106**, 1293—1296).—The slow combustion of tobacco leaves in presence of air and moisture is at first mainly due to the action of microbes. If the tobacco is sterilised, the quantity of carbonic anhydride formed is very small. At 50° the action of the microbes ceases, and oxidation, which takes place above this temperature, is the result of purely chemical changes. C. H. B.

Atmospheric Nitrogen and its Relations to Vegetation. By E. Chevreul (*Compt. rend.*, **106**, 1460—1461).—In 1854 a commission, consisting of Dumas, Regnault, Decaisne, Peligot, and Chevreul confirmed a statement by G. Ville to the effect that living plants absorb nitrogen directly from the atmosphere. All subsequent researches, including those of Gautier and Drouin (this vol., pp. 746, 871), afford further confirmation of this conclusion. C. H. B.

Assimilation of Nitrogen by Plants. By Wilfarth (*Ann. Agronom.*, **14**, 231—233).—The experiments now reported completely confirm the conclusions arrived at by the author and Hellriegel last year (this vol., p. 742). Oats, buckwheat, colza, peas, sainfoin, and lupins were cultivated in pure sand free from nitrogen. The necessary minerals having been added to this sterile soil, the plants grew normally until the nitrogen stored in the seed was used up. A little garden soil was now rubbed up with water, allowed to subside, and the turbid liquid added to the sterile soil. Each pot contained 4 kilos. sand, and received a quantity of liquid containing 0·3 to 0·7

mgram. of nitrogen. After this addition, the oats, colza, and buckwheat remained undeveloped, but the peas, sainfoin, and lupins rapidly became deep green, and continued to grow vigorously to maturity. The same turbid liquid sterilised by heat had no effect in causing the growth of the leguminous plants. The soil from which the liquid is prepared is a matter of importance. Any soil will answer in the case of peas, but lupins and sainfoin require a soil in which the same plants have been previously grown. 178 experimental pots of plants were grown, and the conclusion is that the papilionaceous plants employed are able to obtain their nitrogen from the atmosphere. The following experiment is cited to prove that the combined nitrogen of the air is not the source of supply:—4 kilos. of the calcined sand, with the requisite minerals, but without nitrogen, were placed in a large flask, and sown with one seed each of oats, buckwheat, and peas. A little soil water was then added, and the flask hermetically sealed, the requisite carbonic anhydride being supplied to the enclosed atmosphere at intervals. The result was the same as with the cultivations in free air; the oat and buckwheat seeds grew only until the nitrogen contained in them was exhausted, whilst the pea grew and furnished 6·55 grams of dry matter containing 0·137 gram nitrogen. Lupins show the absorption of nitrogen better than the other plants. In four experiments when soil water was added to the sand, the total dry weight of crop was 217·28 grams, containing 4·786 grams nitrogen; in four similar experiments without the soil water the total dry weight of crop was only 3·660 grams, containing nitrogen 0·0547 grams.

Whilst it is certain that without soil water (or when this is sterilised) none of the characteristic tubercules will grow on the roots of the papilionaceous plants, and also that they are developed when the soil water is supplied, it cannot yet be asserted that the power of absorbing free nitrogen is dependent on the growth of these tubercules. An editorial note adds that these tubercules can easily be found on the roots of clover, some being as large as a pin's head, and that when bruised in a drop of water, numbers of animated bacteria become visible under the microscope. J. M. H. M.

Liberation of Silver by Living Cells, and its supposed Connection with Hydrogen Peroxide. By T. Bokorny (*Ber.*, **21**, 1848—1850).—The agent in living cells which causes the liberation of silver cannot be hydrogen peroxide, as is stated by Wurster, for the following reasons:—It is destroyed by very dilute acids, precipitated by ammonia, gives the reactions for albumin, cannot be extracted, does not give a blue coloration with starch and potassium iodide, does not react with ferrous sulphate and tannin, and is not injurious to living cells even in large quantities. F. S. K.

Accumulation and Consumption of Glycogen in Fungi. By L. Errera (*Chem. Centr.*, 1888, 252, from *Ber. deut. bot. Ges.*, **5**, 74—76).—The author has satisfied himself that a large number of fungi contain glycogen, the only species in which he could not detect its presence being some of the *Uredineæ*. The author also notices a

complete analogy existing between the deportment of the glycogen of the fungi and the starch of the higher orders of plant life; this being especially noticeable in the *Phallus impudicus.* J. W. L.

Formation of Glycogen in Beer Yeast. By E. LAURENT (*Chem. Centr.*, 1888, 252, from *Ber. deut. bot. Ges.*, **5**, 76—78).—By cultivating yeast in gelatin, the author succeeded in bringing about an accumulation of glycogen by several species of *Ustilagineæ*. The presence of carbohydrates, egg albumin, and many other substances would seem to promote its formation. J. W. L.

Formation of Calcium Oxalate in Leaves. By A. F. W. SCHIMPER (*Ann. Agronom.*, **14**, 175—187, from *Bot. Zeit.*, 1888, Nos. 5—10).—Calcium oxalate is very generally found in leaves, although it seems to be wanting in certain families of plants, such as mosses and most ferns and grasses. In order to observe the distribution of this substance in entire leaves or parts of leaves, the author deprives them of their chlorophyll and intercellular air by alcohol, and then places them for several hours in a solution of 8 parts of chloral hydrate in 5 parts of water. This renders them quite transparent, and the crystals of oxalate may be readily seen on examining them under the microscope with the intervention of Nicol's prisms. Working in this way, the author finds that the *raphides*, or clusters of needles of calcium oxalate, which are only developed in certain plants, are formed while the leaves are very young, and do not increase as the leaf grows. It is quite otherwise with the ordinary crystals of calcium oxalate which are found in the great majority of plants. These are very slightly developed in the young leaves, whereas the old ones are very rich in them. Leaves in full daylight contain more oxalate than those in shade. If any particular leaf is shaded, the oxalate contained will not increase in amount, although it does in neighbouring leaves exposed to the light. A seedling grown in the dark contains very minute crystals of oxalate; these rapidly increase when the plant is brought into the light. The oxalate thus formed is called by the author secondary oxalate; the raphides formed in young shoots of certain plants are called primary oxalate; and there appears to be also a tertiary deposit rapidly formed in autumn in old leaves before their fall.

In the case of variegated plants or leaves, the white portions are found to be almost destitute of secondary oxalate, whilst the green portions contain the normal quantity; petals and other portions destitute of chlorophyll are equally destitute of secondary oxalate. A shoot of *Pelargonium zonale* grown for a month in air deprived of carbonic anhydride developed many leaves of the usual size, but delicate, and showing under the microscope no starch granules, but an abundant crop of oxalate crystals. The formation of secondary oxalate therefore appears to depend on the presence of light and chlorophyll, but to be independent of assimilation; it is greatly promoted by free transpiration. The crystals of oxalate once formed are not stationary, but generally redissolve and migrate as a useless bye product to cells other than those in which they are formed. Thus the oxalate

is generally formed in the assimilating cells, and then migrates to the cells disposed along the veins of the leaves, which are so frequently found filled with crystals. A further migration from the leaf towards the stem seems probable. The formation of the tertiary oxalate deposit has been explained physiologically by the observation of Berthelot and André, that the young leaves contain as much oxalic acid as the old leaves, but that in the former it is present chiefly as potassium oxalate, and in the latter as calcium oxalate, that is, when the activity of the leaf ceases, the oxalic acid is fixed by lime, and the potash liberated to migrate to the still living shoots and leaves.

In order to arrive at the physiological meaning of the secondary deposit of calcium oxalate, the author has endeavoured first to solve the problem why lime is an essential constituent of plants. Various plants, among them *Tradescantia selloi*, were grown in a normal nutritive solution, and in the same solution deprived in succession of calcium, potassium, and magnesium salts, also in solutions containing these metals in the forms of nitrate, sulphate, and phosphate respectively. The development of the *Tradescantia* was normal in all the solutions for the first three weeks, and continued to be so in the complete solution. In the solution without lime, the leaves successively became smaller and smaller, brown and dead in patches, and the buds at last dried up. Under the microscope, the assimilating cells of the leaves were seen to be surcharged with starch, so that assimilation was not prevented. The conducting cells were empty of starch, and the raphides of oxalate, formed at first in the youngest shoots, had disappeared. In the solutions without potassium and magnesium salts the old leaves died, but new shoots and leaves continued to be formed. Under the microscope the leaves were seen to be completely deprived of starch and glucose, so that assimilation had ceased, although the migration of carbohydrates continued until new shoots were formed, the plants eventually dying of exhaustion just as if cultivated in air deprived of carbonic anhydride. The addition of a little potash salt caused assimilation to recommence. The cells of the plant grown in the complete solution showed abundance of raphides and of secondary crystalline deposits of calcium oxalate, and when lime was added to the solution hitherto deprived of it, the plant grown in this solution also began to form these crystals after eight or nine days. It follows from this and other experiments that lime is essential to the migration of carbohydrates, although not to their assimilation, and that, having assisted this migration, perhaps by combining with the soluble carbohydrate, it is afterwards converted into oxalate and thrown out of solution. Secondary deposits of calcium oxalate are formed, whether the calcium be supplied as nitrate, phosphate, or sulphate. By a series of microchemical observations the author shows that these salts enter the leaves unaltered, and are often stored up, especially in the hairs. In the leaves, the nitrogen, phosphorus, and sulphur are assimilated, and the lime removed as secondary oxalate. Even detached leaves (etiolated) will absorb the above-mentioned salts from nutrient solutions and live for several weeks, becoming healthy and increasing in size, and it is easy by microchemical means to follow the disappearance of the nitrate reaction (diphenylamine) and the corresponding

decomposition of crystals of calcium oxalate. This decomposition of nitrates takes place only in light and in green cells; probably the assimilation of sulphur and of phosphorus is dependent on the same conditions. J. M. H. M.

Vanillin in the Seeds of Lupinus albus. By G. CAMPANI and S. GRIMALDI (*Chem. Centr.*, 1888, 377, from *Boll. R. Accad. dei Fisiocritici in Siena*, **5**).—By distilling the lupine seeds six times with steam and evaporating the distillate to one-tenth of its original volume, a liquid is obtained which smells of vanillin, and turns litmus-paper very gradually red, eventually bleaching it. By extracting this liquid with ether, vanillin is obtained and was purified by recrystallising it from water after having boiled it with animal charcoal. Its melting point (81°) and its reactions proved its identity. J. W. L.

Presence of Albumin in Cell-fluid. By O. LOEW and T. BOKORNY (*Chem. Centr.*, 1888, 252—253, from *Bot. Zeit.*, 1887, 849—857).—The authors find active albumin in the cell fluid of a number of spirogyra in which it is dissolved. By the action of bases it is precipitated in a granular condition. It reduces alkaline silver solution and with many reagents behaves like egg-albumin. Since the threads of protoplasm consist of active albumin it appears in this precipitated form in them also. J. W. L.

Morphological and Chemical Composition of Protoplasm. By F. SCHWARTZ (*Ann. Agronom.*, **14**, 229—231).—In the long memoir of which this is an abstract, the author elucidates the chemical properties of the substances which can be distinguished morphologically in the protoplasmic body, and describes microchemical reactions by which they can be identified.

Cell-sap and protoplasm.—The cell-sap is sometimes acid, sometimes alkaline. Sometimes it naturally contains a substance sensitive to change of reaction, and therefore serving as indicator; when this is not the case the substance is placed in a solution of red cabbage which is then traversed by an electric current. Under this influence the cabbage solution penetrates the protoplasm and shows its reaction, as well as that of the cell-sap. Protoplasm is never acid, most frequently it is distinctly alkaline. The chromatophores, microsomes, and in some cases the proteïc granules, show the same reaction. Probably the alkalinity is due to alkaline phosphates.

Chloroplastides.—The plasmatic substratum of the chlorophyll grains consists of two substances, *chloroplastin* and *metaxin*, the first forming sinuous fibrils, sometimes uniformly green, sometimes charged with little spherules more deeply-coloured. Between the fibrils is the colourless metaxin, hardly visible in the intact chloroplastides. When the chlorophyll granules are treated with water, the chloroplastin is not affected, the metaxin first swells and then dissolves. It is the latter substance which causes the formation of vacuoles in grains of altered chlorophyll.

Nucleus.—Five different substances are contained in the nucleus, two of them, *chromatin* and *linin*, belonging to the framework, the

first forming the microsomes, the second the nucleohyaloplasma of Strasburger.

The nucleole consists of *pyrenin*, which is quite distinct from chromatin. The substance constituting the membrane of the nucleus and resembling pyrenin in most of its reactions, receives the name of *amphipyrenin*. The liquid contents of the nucleus, or nucleochyme of Strasburger, is similar to linin, and is called paralinin. Cytoplasma, or general plasma of the cell, presents very rarely a reticulated or fibrillar structure. It is essentially formed of *cytoplastin*, the only constant proteïc substance of protoplasm, which furnishes equally the internal and external membrane of the protoplasmic body. Then come the substances dissolved in the cell-cap or those forming the microsomes suspended in the cytoplastin.

Chloroplastin and cytoplastin are insoluble in concentrated potassium hydrate and in sodium chloride (1 : 10), in which the nucleus dissolves completely. They resist the action of trypsin and pepsin, whilst the substances of the nucleus are digested, pyrenin with difficulty. In hydrochloric acid (1 : 100) chloroplastin swells strongly, cytoplastin is precipitated; in disodium phosphate (5 : 100) the former is insoluble, the latter swells and dissolves. Chromatin and pyrenin fix colouring matters strongly. Pyrenin is insoluble in sodium chloride (20 : 100), saturated solution of magnesium sulphate, acid potassium phosphate (1·5 : 100), potassium ferrocyanide acidified with acetic acid, and copper sulphate. These reagents dissolve chromatin. Chromatin is insoluble and pyrenin soluble (after swelling) in acetic acid (3 : 100) and hydrochloric acid (1 : 100). Amphipyrenin does not fix colouring matters, dissolves less easily than pyrenin in sodium chloride (10 : 100), and more easily in potassium hydroxide (0·1 : 100). Linin and paralinin swell strongly in sodium chloride (20 : 100), lime-water, strong potassium dichromate, and often in water only, and in sodium phosphate (1 : 100), media in which amphipyrenin is insoluble. Solution of magnesium sulphate causes paralinin to swell and does not alter linin; pepsin digests paralinin but not linin. Metaxin, unlike the plastins, is digested by pepsin and trypsin, and is distinguished from the substances of the nucleus by its solubility (after swelling) in hydrochloric acid (0·1 : 100). J. M. H. M.

Constituents of Calamus Root. By H. Thoms (*Ber.*, **21**, 1912—1920).—Calamus root was extracted with light petroleum to remove the oil and then treated with alcohol which extracted a sugar and tannic acid. The root, after this treatment, has no odour, is no longer bitter and has a woody taste. If it is now treated with water an extract is obtained containing potassium and sodium sulphates, an acid salt of tartaric acid, and dextrin.

The sugar of calamus root is dextrose.

Acorin forms a thick, honey-coloured substance of an aromatic odour and bitter taste; it contains no nitrogen and is neutral to litmus. When treated with dilute acid it is decomposed into an ethereal oil, a resin acid, and what is probably a sugar.

When a calamus extract is heated with alkali, trimethylamine is formed (compare Geuther, Abstr., 1887, 972). N. H. M.

Influence of Sterilisation of Soil on the Growth of Plants.—By TSCHIRCH (*Ann. Agronom.*, **14**, 233—234).—The roots of the beech tree are always enveloped in a layer formed by the interlacing hyphæ of a fungus. If comparative cultivations be made in ordinary forest soil and in the same soil sterilised, it is easily seen that sterilisation has a noxious influence. 10 out of 15 plants died when so treated, whilst all the 15 planted in the unsterilised soil grew and flourished and their roots became covered with the fungus. The author contrasts this with ordinary agricultural crops which do not live in symbiosis with a fungus, and which flourish better in sterilised soil than in soil containing micro-organisms. He asserts that this is true of lupins also, and hence that the root tubercules cannot be of importance to this plant when growing in ordinary arable soil. It does not precisely follow from this that Hellriegel's view is incorrect, since his experiments were made in a soil free from nitrogen, whilst the author's were made in ordinary soil, perhaps containing abundance of ready-formed nitrate. It is still possible, therefore, that these tubercules play an important part in the assimilation of atmospheric nitrogen by the leguminosæ. J. M. H. M.

Absorption Compounds and the Absorptive Power of the Soil. By J. M. VAN BEMMELEN (*Landw. Versuchs-Stat.*, **35**, 69—136).—The author first gives an account of the results of his own investigations and those of other observers on the absorptive power of colloïd substances for acids, bases, and salts. He studied particularly the gelatinous hydrates of the dioxides of silicon, tin, and manganese, and of some other oxides. These gelatinous hydrates, for which he adopts Graham's name—"hydrogel," when obtained free from apparent moisture, still retain an amount of water varying with the conditions under which they have been dried and which does not correspond with any definite chemical formula. Hydrogels can form absorption-compounds with gases and liquids, and can also, by absorption, remove acids, bases, salts, &c., from solutions with which they are brought into contact. The formation of these absorption-compounds must be ascribed to something more than adhesion, although they cannot be considered as ordinary chemical compounds. When a hydrogel is brought into contact with a solution, the dissolved substance is absorbed by the hydrogel until a state of equilibrium is reached, the tendency of the hydrogel to absorb more of the substance being counterbalanced by the tendency of the solvent to remove the substance from the hydrogel. This state of equilibrium will be altered if the conditions change; such as the temperature, the strength and relative amount of the solution.

The principal phenomena of absorption are as follows:—

1. When a quantity of an absorption-compound gains or loses some of the absorbed substance, in all probability every particle becomes equally richer or equally poorer in the absorbed substance. In this respect such compounds differ from chemical compounds, for when these suffer partial dissociation a certain number of the molecules are completely decomposed and the rest remain intact.

2. The absorptive power is not constant, but varies as absorption

goes on; the attraction for the first portions absorbed is strong and the absorption goes on rapidly; but as more and more is absorbed the tendency to absorb more becomes weaker and absorption takes place more slowly. Conversely, in removing absorbed substances, the first portions are given up readily, and the last portions are retained much more obstinately.

3. The mode of formation and also the drying, warming, &c., of colloïd substances cause an alteration in them and change their absorptive power.

4. Hydrogels may change into ordinary chemical hydrates, acquiring a definite composition, and sometimes a crystalline form; but in doing so they lose their power of forming absorption-compounds. The formation of a colloïd compound is accompanied by the evolution of considerably less heat than the formation of the corresponding crystalline compound.

5. Temperature affects the absorptive power. Increase of temperature sets free a certain quantity of water from the hydrogel, and also increases the solvent action of the water or the substance absorbed, consequently it causes a diminution in the amount absorbed.

6. Every hydrogel has its own specific absorptive power for each acid, base, and salt. One hydrogel may absorb acids more powerfully than it does other substances, another bases, and a third salts. In general absorption is strongest when under other circumstances the hydrogel and the absorbed substance can combine chemically; for instance, stannic acid absorbs much sulphuric acid, and still more potash.

The substance absorbed may be merely proportionally divided between the water of the hydrogel and the water of the solution, as is the case when potassium chloride is absorbed by the hydrogel of silica; or else a larger proportion than this may be absorbed by the hydrogel, for instance, on separating the hydrogel of metastannic acid from a dilute potash solution, the filtrate is nearly free from potash. The absorption may even cause decomposition of the substance in solution; thus the hydrogel of silica removes some potash from potassium carbonate or some soda from disodium phosphate, leaving a corresponding amount of potassium bicarbonate or monosodium phosphate in solution; also on shaking up the same hydrogel with calcium carbonate and potassium chloride, there is an absorption of lime and potash, corresponding amounts of calcium chloride and calcium bicarbonate being left in the solution, and some potassium chloride is also absorbed.

7. Its condition and its weight being given, the temperature also being given and remaining constant, and the hydrogel not being soluble in the liquid, then the amount of a particular substance absorbed by it varies with the state of concentration and with the amount of the solution. A state of equilibrium is arrived at between the absorptive action of the hydrogel on the one hand and the opposing action of the water on the other; if, however, chemical decomposition of the substance occurs, then the attraction of chemical combination takes a part in producing the equilibrium.

The stronger the solution is the more of the substance is absorbed,

but in a decreasing proportion, and the limit is reached when after the equilibrium point has been attained, the liquid is in a state of saturation.

However much the quantity and however much the state of concentration of the solution may vary in different experiments, yet as soon as equilibrium is reached, according as the final strength of the solution in one experiment is greater or less than in another experiment, then is the amount absorbed also greater or less; and, further, if the final strengths of the solutions are the same in two experiments then also the amounts absorbed are the same; and, conversely, if the amounts absorbed in two experiments are the same, then also are the final strengths of the solutions the same.

Hence a curve can be constructed for a particular hydrogel and a particular substance representing the relation between the amount of substance absorbed and the final strength of the solution.

The author discusses the question whether a mathematical expression can be given for the formation of absorption-compounds. In the case of dilute solutions and provided the absorptive power of the colloïd is weak, as is the case with silicic acid and with soil acting on saline solutions, the curve is nearly a straight line, and an expression can be given which is approximately correct. But in other cases no satisfactory formula can as yet be found.

8. One crystalloïd absorbed by a hydrogel can be replaced by others. If a hydrogel which has absorbed a substance A be placed in a solution of another substance B, then the solvent will dissolve out some of A, and at the same time the hydrogel will absorb some of B until equilibrium is attained in each case. No true substitution has here taken place. As long as the absorbed quantities are small A and B are absorbed without mutually influencing one another to a noticeable extent; but if the quantity of A is large it may happen that not only does loss of A happen as above, but there may also be a further loss of A owing to its replacement by B, for since A is largely absorbed, part of it is not strongly fixed by the hydrogel, and this part may be replaced by a part of B which the hydrogel fixes the more strongly the smaller the total quantity absorbed of B is. This is a true substitution. By repeatedly treating the hydrogel which has absorbed A with solutions of B, A can be entirely replaced by B. If chemical action occurs between A and B the phenomena are more complicated.

If absorption occurs by chemical substitution, for instance, when the colloïd silicates of the soil absorb potash from a potassium chloride solution, in exchange for lime, then the law given under (7) holds good, namely, that a relation exists between the amount absorbed and the final strength of the solution, whatever the proportions and strengths at starting were, and that thus, other things being the same, the absorption depends only on the final strength of the solution.

The author next treats of the absorptive power of the soil. This is chiefly due to the fine particles, and these are mainly composed of colloïd substances. The colloïd components of the soil are (1) the substances classed as humus; (2) ferric and ferrous hydrates; (3) silicic acid; (4) the amorphous silicates produced by weathering and resembling zeolites.

All attempts that have been made to isolate individual members of the humus-group and assign formulæ to them have failed, for they have been formed from colloïds and are of a colloïd nature themselves. Soluble humus substances form absorption-compounds with acids and salts, but most easily with bases, hence they were termed acids. The soluble humus acids, in the stage of the so-called crenic and apocrenic acids, can form soluble complex compounds with ammonia, with alkalis, and also with different insoluble bases such as lime, magnesia, ferrous and ferric oxides, &c. This is how it is that brown peat-water in nature contains traces of iron, and that the hydrochloric acid extract of a clayey peat soil (free from excess of hydrochloric acid) often gives no precipitate of ferric or aluminium hydrates on the addition of ammonia.

Humus as it exists in the soil contains ammonia, alkalis, alkaline earths, and ferrous oxide; if this humus is treated with solutions of salts, absorption by exchange of bases will take place; thus on treatment with potassium chloride solution, potash will be absorbed, and lime and magnesia will go into solution. By extracting humus with dilute acid, the bases are removed and this kind of absorptive power is reduced to a minimum.

Peat humus has a strong absorptive power for alkalis, and especially for ammonia. When peat humus is treated with solution of a caustic alkali, part is dissolved, but the undissolved part absorbs much alkali. This kind of absorptive power is not lost after extraction with hydrochloric acid. Humus also removes alkalis from solutions of their salts with feeble acids. When humus is treated with a phosphate of an alkali the alkali is absorbed, turning out some lime and magnesia which precipitate some phosphoric acid and thus cause its apparent absorption; but if the humus contains little or no lime or magnesia this apparent absorption of phosphoric acid does not take place.

Humus substances probably possess some small absorptive power for whole salts.

Hydrated iron oxides and hydrated silicic acid rarely occur free in the soil to any great extent.

(4.) To the zeolite-like silicates is due the peculiar nature of clay soils. They have the property of drying to hard, compact masses, and also of remaining for a long time in suspension in pure water, but when in suspension, small quantities of acids, bases, and salts cause them to collect in flocks and to subside rapidly. The granular structure of a clay soil is chiefly due to the soluble salts, especially to calcium bicarbonate; the bases and salts absorbed by the colloïds must also exert an influence. If the soluble salts are removed, the structure is altered and the clay becomes a mud.

These silicates are probably complex compounds of various colloïd silicates of aluminium, iron, the alkalis, and the alkaline earths, which contain also alkalis and alkaline earths in a state of absorption, but it is not possible to make out the exact relationship of the constituents. They can absorb free alkalis, alkaline earths, and ammonia from solutions, and they can also absorb bases by replacement from solutions of their salts. Potash is most strongly fixed by the soil,

and, consequently, is most strongly absorbed from saline solutions, replacing equivalent quantities of lime, soda, and magnesia. If a soil is treated with solutions of a potash, magnesia, soda or lime salt containing like amounts reckoning in equivalents, then the final strength of the potash-salt solution is less than that of the magnesia-salt solution, and that of the magnesia-salt solution is less than that of the soda- and lime-salt solutions, for the action of a dissolved lime, &c., salt on the absorbed potash is weaker than the action of a dissolved potash salt on the absorbed lime.

If a soil has been saturated with potash, &c., this can be replaced by repeated treatment with solution of another salt, for the potash, &c., extracted each time is removed and can exert no reversing action, and, further, each time the solution is weaker in potash and the reversing action is therefore less.

That soda salts promote an apparent absorption of phosphoric acid from a phosphate solution is explained by soda replacing lime and magnesia, which then produce a precipitate of calcium and magnesium phosphates.

Treatment of a soil with strong hydrochloric acid decomposes, to a great extent, those silicates to which the absorption, accompanied by exchange of bases, is due, although the soil still retains much aluminium silicate and aluminium potassium sodium silicate.

In addition to absorption by exchange of bases, the colloïd silicates can absorb the alkalis and alkaline earths as wholes, and they can also absorb salts as wholes, though their power to do the latter is feeble.

The conditions of absorption as it occurs in the soil in nature are not exactly the same as in experiments; for example, in nature the final equilibrium is rarely reached; and in experiments the soil is shaken with proportionally large volumes and proportionally concentrated solutions. The results of absorption experiments are only of limited value as a guide to the fertility of a soil, and must be considered in connection with the results of analysis; they cannot supersede the latter. H. H. R.

Rapid Fermentation of Grape-juice. By A. Audoynaud (*Ann. Agronom.*, **14**, 211—221).—Many salts added to the juice in small proportion (0·5—1 gram per kilo. of fresh grapes) increase the vitality of the yeast. Most active are ammonium sulphate, phosphate, and carbonate, probably because they supply nitrogen to the yeast plant in a more available form than the albuminoïds of the must. Nitrates cannot be substituted. Soluble calcium salts, such as the nitrate, sulphate, and bicalcic phosphate, also hasten the fermentation. Magnesium salts are far less efficacious. Boric acid is without action. The proportion of water has considerable influence on the rapidity of fermentation. The following conditions ensure great rapidity and regularity; water seven to eight times the weight of the sugar present; temperature 25°; ammonium carbonate and bicalcium phosphate each added in the proportion of 500 grams per 1000 kilos. of grapes; the must must remain acid after all these additions.

J. M. H. M.

Effect of Nitrogenous Manures on Tobacco. By C. O. Harz (*Ann. Agronom.*, **14**, 234—235, from *Bot. Centralbl.*, **33**, 218).—From two years' experiments on small plots the author concludes that sun is more favourable than shade to the amount of crop and to its richness in nicotine, and that ammonium sulphate is better than an equivalent quantity of sodium nitrate. J. M. H. M.

Organic Nitrogenous Manures. By A. Petermann (*Bied. Centr.*, 1888, 366—367).—The percentage of nitrogen in waste wool, blood and leather meal, which were used in experiments on roots during 1885, 1886, and 1887, are here given, as also that contained in various cakes used as manures. The percentage of sugar in the roots as affected by the manures is also stated. E. W. P.

Analytical Chemistry.

Improved Wash-bottle. By J. Sobieczky and V. Hölbling (*Zeit. anal. Chem.*, **27**, 365—366).—To prevent the retreat of the liquid from the point of the jet, and the consequent forcible ejection of the first drop when recommencing to blow into the bottle, a small Bunsen valve, made with very thin caoutchouc tube, is attached to the swan-neck tube in such a way as to lie within the rubber connection which gives flexibility to the jet. M. J. S.

Different Methods of Estimating Chlorides in Urine. By Brignone (*Chem. Centr.*, 1888, 808, from *Ann. Chim. Farm.*, **7**, 137—159).—The different methods of estimating chlorine in urine, both directly and after destruction of the organic matter, are fully discussed. The author gives the preference to those processes in which the organic matter is previously destroyed by fusion with nitre. A process is also mentioned in which the organic matter is oxidised with potassium permanganate, and the titration of chlorine made directly with silver nitrate and potassium chromate; sulphates and nitrates act injuriously. Denigés' process, which seems to be the same as the above, gives higher results than any other method. The results are, however, sufficiently accurate for clinical purposes, and the process is simple to carry out. J. P. L.

Determination of Nitrogen by Soda-lime. By W. O. Atwater (*Amer. Chem. J.*, **10**, 197—209).—It is unnecessary to powder the substance beyond allowing it to pass through a 1 mm. mesh. No loss of nitrogen by formation of alkaline cyanides need be feared if the soda-lime contain enough water and other conditions mentioned be adhered to, neither is there any loss of nitrogen gas except by oxidation of the ammonia. The most important error is caused by the formation of volatile nitrogenous compounds that pass through the

tube unchanged, and are not estimated either by standard acid or by platinum chloride; it is eliminated in most cases by packing the tube as described (this vol., p. 752), but in other cases where leucine-derivatives are dealt with or quinoline-derivatives are volatilised, these precautions are totally inadequate. The formation of red coloured substances or of a turbidity in the absorbing acid is no proof that the result is inaccurate. H. B.

Estimation of Total Nitrogen in Organic Compounds. By P. Cazeneuve and L. Hugounenq (*J. Pharm.* [5], **17**, 545—547, 593—594).—The authors modify Dupré's improvement of Dumas' method, and proceed as follows:—At the bottom of a combustion tube 15 cm. long is placed some dry manganese carbonate. This source of carbonic anhydride is preferred as it can be easily dried, its decomposition is indicated by change of colour, and on cooling it does not reabsorb carbonic anhydride. The remainder of the tube is charged as usual, and connected with a pump and manometer. To remove the air, the tube is exhausted three times successively, and each time carbonic anhydride is expelled from the manganese carbonate until the mercury descends completely; then, Dupré's apparatus is applied whilst the carbonic anhydride is still being evolved, and the combustion proceeded with in the ordinary way. Urine is very satisfactorily treated by mixing 5 c.c. with 5 grams of dry plaster of Paris as indicated by Washburne, together with 0·05 gram oxalic acid. After drying on the water-bath, the mass is intimately mixed with 30 grams of copper oxide and burned as above. J. T.

Determination of Ammonia in Urine. By C. Wurster (*Zeit. anal. Chem.*, **27**, 406—407, from *Centr. Physiol.*, 1887, 485).—The mixture of the urine with baryta-water is placed in a flask, the bottom of which just touches a water-bath kept at 50°. At this temperature urea is not decomposed by baryta. The flask is connected with a second which plunges completely into the water of the bath and serves for arresting froth. This is followed by a bulb absorption apparatus, containing titrated acid, standing in cold water, and connected with the air-pump. About two-thirds of the mixture is distilled over whilst working the pump. Air is admitted to the overflow flask before disconnecting, and the residual acid is titrated.

M. J. S.

Estimation of Phosphoric Acid in Basic Slag. By J. H. Vogel (*Chem. Zeit.*, **12**, 85—86).—The author recommends the following modified form of Müller's method of estimating phosphoric acid in basic slag, as more convenient and quicker than the original. 5 grams of the slag is dissolved by heating for half an hour in a half-litre flask with 40 c.c. of water and 40 c.c. of hydrochloric acid, sp. gr. 1·12 (compare Kennepohl, this vol., p. 321), it is then filled to the mark and filtered. 50 c.c. of the solution is mixed with 10 c.c. of a 50 per cent. solution of citric acid, nearly neutralised with 10 per cent. ammonia, 20 c.c. of magnesia mixture gradually (during two minutes) dropped and stirred in, finally 10 to 12 c.c. of concentrated ammonia is added and the whole shaken round for a minute. As a

rule after two hours the solution may be filtered and proceeded with in the usual manner. D. A. L.

Estimation of Manganese in Pig-iron, Steel, &c. By G. Weissmann (*Chem. Zeit.*, **12**, 205).—Manganese may be accurately determined in iron, &c., in the following manner:—0·4 gram of borings is dissolved in 30 to 40 c.c. of a solution, consisting of 10 parts of concentrated nitric acid, 10 parts of water, and 2 parts of concentrated sulphuric acid; 4 to 5 grams of chemically pure lead oxide is then added with vigorous shaking, the whole boiled for two or three minutes, and this operation again repeated. After settling, the permanganic acid is filtered through ignited asbestos and titrated with ammonium ferrous sulphate solution and dilute permanganate (1 gram in a litre). An estimation occupies at the most an hour, and concordant results have been obtained by this method with irons containing 2 to 3 per cent. of managnese. The operations should succeed one another without intermission, and should be conducted in a room not exposed to sunlight. D. A. L.

Estimation of Lead in Tin Alloys. By Y. Schwartz (*Chem. Zeit.*, **12**, 52).—One gram of the lead-tin alloy, rolled moderately thin, is warmed gently with 20 c.c. of hydrochloric acid until action ceases, which takes about half-an-hour; without removing any undissolved metal, bromine is added until solution is complete, and the liquid yellow in colour. The excess of bromine is boiled off, the solution diluted to 100 c.c., allowed to cool, and then poured in a thin stream, which is constantly moved about, into a solution of 40 grams of commercial sodium sulphide in 150 c.c. of water. When the lead sulphide has settled, it is separated, washed with dilute ammonium sulphide (containing 1 vol. ammonium sulphide prepared from commercial 10 per cent. ammonia to 9 vols. of water), converted into sulphate, and weighed as such. The lead sulphate is then dissolved in basic ammonium tartrate, and any insoluble tin oxide is collected and allowed for. D. A. L.

Separation and Estimation of Antimony and Tin. By Loviton (*J. Pharm.* [5], **17**, 361—363).—Antimony can be completely precipitated as sulphide in a solution containing equal volumes of water and hydrochloric acid of 22° B., whilst the precipitation of tin begins only in a solution containing at least three volumes of water to one of acid. This is used qualitatively by dissolving the alloy in concentrated hydrochloric acid with the addition of a few drops of nitric acid or a little potassium chlorate, then diluting the solution so that only the antimony is precipitated by hydrogen sulphide. The antimony sulphide rapidly settles, and the liquid is filtered through glass-wool, as paper will not withstand the strong acid, and the precipitate is washed with a liquid formed of one volume hydrochloric acid of 22° B., and one and a half volumes of hydrogen sulphide water. The filtrates are sufficiently diluted, and hydrogen sulphide is passed through to complete the precipitation of the tin. The separated metals can be estimated by dissolving in a freshly prepared solution

of sodium sulphide, and electrolysing in Riche's apparatus with a couple of Bunsen cells. This method of separation seems to be applicable to some other members of the group of metals precipitable by hydrogen sulphide. J. T.

Application of Diazo-compounds to the Detection of Organic Matter in Water. By P. GRIESS (*Ber.*, **21**, 1830—1832).—Very small quantities of organic substances present in water can often be detected by means of diazo-compounds, of which diazobenzene sulphate is the most suitable. Two to four drops of a freshly prepared 1 per cent. solution of diazobenzene sulphate, made slightly alkaline with soda, are added to the sample of water placed in a colourless glass cylinder, and the mixture stirred. If no change of colour occurs within five minutes, the almost complete absence of human or animal excreta or putrefying matter may be inferred, but a yellow coloration shows the presence of larger or smaller quantities of such impurities. One part of human urine in 5000 parts of water and 1 part of horse's urine in 50,000 parts of water can be recognised with certainty. Good spring water gives no coloration, but when 100 c.c. of such water is mixed with several drops of waste water from a town canal, the reaction is, as a rule, at once perceptible. 100 c.c. of water, containing $\frac{1}{10}$ c.c. of normal human urine, gives, with this reagent, the same shade as is produced by Nessler's solution in an equal quantity of water containing $\frac{1}{20}$ mgram. of ammonia.

Substances containing sugar, even when present in relatively large quantities, cannot be detected by this method with any degree of certainty. Diabetic urine, containing 6 per cent. of grape-sugar, gave, after suitable dilution, a far less intense coloration than an equal quantity of normal human urine. F. S. K.

Incineration of Organic Substances. By H. KRONBERG (*Chem. Zeit.*, **11**, 1600—1601).—For substances that swell up or explode, or fall to powder when heated, the author recommends burning a small portion of the substances in a covered platinum crucible over a bunsen, breaking up the burnt mass with a piece of platinum wire, and when moderately cool adding a further portion of the substance, repeating the operations until sufficient substance has been burnt. The incineration is complete in the manner recommended by Kobrich (this vol., p. 325). The platinum wire used as a stirrer is weighed with the crucible and contents, a slit is made in the side of the crucible lid for its support. D. A. L.

Detection of Impurities in Alcohols. By X. ROCQUES (*Compt. rend.*, **106**, 1296—1298).—If pure benzene is used in Godefroy's process (this vol., p. 875), the results are not sensibly different from those obtained by Savalle's test with sulphuric acid alone. If, however, the so-called pure benzene of commerce is employed, a coloration is obtained even with pure alcohol, and in presence of aldehydes the coloration is much darker than with sulphuric acid alone. The author concludes that the addition of pure benzene does not increase the delicacy of the action of sulphuric acid, the results obtained by

Godefroy being due to the presence of impurities in the benzene used. In the absence of aldehydes, the addition of benzene does not increase the efficiency of sulphuric acid as a reagent for the detection of the higher alcohols. C. H. B.

Behaviour of Catechol with Fehling's Solution. By A. Wohl (*Chem. Centr.*, 1888, 278, from *Deut. Zuckerind*, **13**, 37—38).—In presence of a large excess of sugar, catechol appears not to interfere with the accuracy of the Fehling's method, in fact, 0·2 per cent. may be present without its causing any serious error. A much larger quantity of catechol appears to decrease the quantity of cuprous oxide precipitated, and the author suggests that the catechol acts the part of an oxygen carrier from the air, it having the property of absorbing free oxygen in a marked degree. J. W. L.

Supposed Reaction of Phloroglucinol. By P. Cazeneuve and L. Hugounenq (*Bull. Soc. Chim.*, **49**, 339—341).—The test for phloroglucinol given in many works, which consists in adding a solution of potassium nitrite to a mixture of aniline or orthotoluidine nitrate and a very weak solution of phloroglucinol, is useless as a qualitative reaction for the latter. The same, or a very similar reaction, is given by phenol, resorcinol, orcinol, α-naphthol and β-naphthol, in 0·05 per cent. aqueous solution. F. S. K.

Determination of Sugar by Alcoholic Fermentation. By M. Jodlbauer (*Chem. Centr.*, 1888, 769, from *Zeit. Rüb-Zuck. Ind.*, **25**, 308—346).—In the present paper the author considers the practicability of estimating sugar by alcoholic fermentation. He finds that under certain conditions the products are constant, and these conditions are—(1.) The use of well-developed yeast taken from an active fermentation, and consequently free from metamorphic changes in its protoplasm. (2.) Maintenance of a definite relation between the amount of yeast and the amount of sugar; the yeast should not exceed 50 per cent. of the amount of sugar. (3.) Exclusion of free oxygen. (4.) The use of a nutrient solution suitable to the full development of the yeast, so that the latter may during fermentation repair its waste. Unless the cell is well nourished, decomposition is slow and incomplete. The most favourable temperature is 34°; the most favourable concentration, 8 per cent.

Of the fermentation products, carbonic anhydride is the simplest to determine, and also affords the most trustworthy results. Cane-sugar and anhydrous maltose give 49·04 per cent.; dextrose, 46·54 per cent. of carbonic anhydride. The duration of the fermentation is of course dependent on the variety of sugar. Cane-sugar requires twice the time that maltose and dextrose do. J. P. L.

Estimation of Sugar in Beet. By A. Petermann and others (*Bied. Centr.*, 1888, 408—412).—Petermann, having carefully examined all the best methods in use, states that (1) the rotatory power of saccharose is the same whether the solvent be water or 60 per cent. alcohol; (2) 85 per cent. alcohol removes all sugar from a well-made mash of

roots; (3) a solution of sugar in 60 per cent. alcohol is not decomposed at the boiling point of the solution; (4) 25 to 50 grams of a well-made mash may be considered as a sufficient quantity in which to estimate the average percentage of sugar; (5) those methods which depend on the mixing of beet-juice with a like volume of absolute alcohol, give results identical with those obtained by the ordinary methods; and as cold alcohol does not precipitate the non-saccharine matters, this modification has no advantage over the old methods; (6) the percentage of sugar obtained by digesting or extracting the mash with alcohol is, on the average, 0·5 per cent. lower than that obtained by the old process of polarisation; the causes of this error are that the analysed juice is not the natural or normal juice, non-saccharine matters are present, and the correcting factor 0·95 is incorrect. Pellet, however, comes to a conclusion opposite to that of Petermann, for he recommends the digestion with water as the most correct, convenient, and cheapest, and he considers that even after the alcohol has been in contact with the mash for half an hour, there is no certainty that all the sugar has been extracted, in fact he finds that 0·4 to 0·7 per cent. less is extracted than when water is employed, and when digestion has been allowed for an hour, the amount extracted is the same as when digestion with water has proceeded for only half an hour. Pellet recommends the following procedure: 52·1 grams of the mash for the German polariser, and 32·4 for the French, is introduced into a flask marked at 200 and 210 c.c., the interval between being graduated to 0·5 c.c., then 175 c.c. of water and 5 c.c. of lead acetate are added, the flask filled up to 200 c.c. with water, and heated for half an hour at 85—90°, then cooled, filled up to 201·5 c.c., shaken, and filtered. E. W. P.

Reduction of a Solution of Methyl-violet by Invert Sugar. By A. Wohl (*Chem. Centr.*, 1888, 739, from *Zeit. Rüb-Zuck. Ind.*, **25**, 347—352).—This test for minimal quantities of invert sugar employed by Ihl (this vol., p. 876) has been submitted to further experiment. It is found that the time required for decolorising the solution becomes longer as the solution becomes weaker, and that it is also dependent on the alkalinity of the solution. In order to obtain comparable results, and an approximation of the amount of invert sugar present, it is expedient to maintain the alkalinity constant, which is too tedious for practical purposes. Other reducing substances which occur in raw sugar, and which are not precipitated by lead acetate, do not decolorise the solution during the short time it is boiled. Ammonium salts hinder or delay the reaction. J. P. L.

Estimation of Small Quantities of Sugar in Urine. By A. Pollatschek (*Chem. Centr.*, 1888, 768—769, from *Deut. Med. Wochensch.*, **14**, 354—355).—The reaction with Fehling's solution is much clearer and sharper if the urine is first shaken with good animal charcoal (prepared with hydrochloric acid) and filtered. The substances which hinder the reaction are thereby removed. As a control the delicate phenylhydrazine test should be used.

J. P. L.

Detection of "Saccharin." By C. SCHMITT (*Zeit. anal. Chem.*, **27**, 396—397, from *Rep. anal. Chem.*, **7**, 37).—For the detection of Fahlberg's "saccharin" by converting it into salicylic acid, it is necessary to ensure the absence of tannin, since that substance also yields traces of salicylic acid when fused with sodium hydroxide. The insolubility of tannin in a mixture of equal parts of ether and light petroleum furnishes a convenient method. If salicylic acid is present, its amount may be colorimetrically determined and allowed for. For detecting saccharin in wine, 100 c.c. is strongly acidified, shaken three times with a mixture of ether and light petroleum, the ethereal extract mixed with soda solution (not potash), evaporated in a silver basin, and the residue fused with more soda for half an hour at 250°. The melt is dissolved in water, acidified with sulphuric acid, and shaken with ether. The residue from the evaporation of the ether is tested with ferric chloride for salicylic acid. M. J. S.

Modification of the Methyl-violet Reaction for the Detection of Free Hydrochloric Acid in Gastric Juice. By G. KOST (*Chem. Centr.*, 1888, 731, from *Centr. Klin. Med.*, **9**, 243—244).—Penzoldt found that peptone and albumin do not hinder the methyl-violet reaction if they are first precipitated with a 10 per cent. solution of tannin. The author has been enabled to corroborate this result in a number of cases of cancer of the stomach. For the diagnosis of carcinoma of the stomach, however, the modified process does not appear to be so trustworthy as the original one. It is at present chiefly of theoretical interest, as it has from another source led to a confirmation of Cahn and von Mering's statements regarding the uncertainty of the methyl-violet test, and the frequent occurrence of free hydrochloric acid in carcinoma of the stomach. J. P. L.

Behaviour of some Acids towards Chromic Acid and Permanganate. By T. SALZER (*Ber.*, **21**, 1910—1911).—When a solution of citric acid is coloured pale-yellow by a drop of potassium chromate solution, the colour remains unchanged, even after addition of sulphuric acid, for some days. A solution of tartaric acid similarly treated becomes colourless. The presence or absence of $\frac{1}{2}$ per cent. of tartaric in citric acid can be detected by extending the experiments over some hours.

Formic, acetic, succinic, and benzoic acids behave like citric acid, whilst lactic acid decolorises the chromate. Tannin and pyrogallic acid produce dark colorations, and phenol and salicylic acid give orange-red colorations.

A solution of 0·1 gram of citric acid and 0·5 gram of sodium carbonate in 20 c.c. of water when treated with a drop of a solution of potassium permanganate (1 : 500) remains coloured; if 0·1 gram of tartaric acid is treated in a similar manner, the solution becomes colourless in 5 to 10 minutes (compare v. Baeyer, *Annalen*, **245**, 103). N. H. M.

University of Michigan

General and Physical Chemistry.

Refractive Indices of the Metals. By A. KUNDT (*Ann. Phys. Chem.* [2], **34**, 469—489).—Determinations of the refractive indices of various metals, and more especially silver, have been made by several observers, but the results obtained are not by any means concordant. Quincke made use of various interference methods, and Wernicke of an absorptive method; Quincke's different determinations gave very different results, and whilst Wernicke gives a value between 3 and 5 for the refractive index of silver, Voigt concludes from the same experiments of Wernicke's, that its value is about 0·25 (Voigt, *Ann. Phys. Chem.* [2], **23**, 104 and 2595). Beer (*Ann. Phys. Chem.*, **92**, 402) and Voigt, by the aid of both Cauchy's and Voigt's theories, have also calculated the velocity of light in various metals from reflection experiments, and in each case they came to the conclusion that Snellius's law of refraction did not hold; the author, however, retains the definition of the index of refraction as the ratio of the velocity of light in a vacuum to that in the metal. The results obtained by Beer and by Voigt agree fairly well, and they both find that the index of refraction is less than unity for silver and gold, approximately equal to unity in the case of copper, and greater for other metals.

All these observers made use of indirect methods, but the author having overcome the difficulty of obtaining prisms of the various metals sufficiently thin to be transparent, measures the index directly from the refraction of the light traversing these prisms.

The basis of each prism consisted of a thin platinised glass plate, the platinum deposit being so uniform that no variation could be detected even under a high magnifying power. The metallic prisms were formed on this in most cases by electrolytic deposition, the method adopted being to place a thin sheet of the required metal perpendicular to but not quite touching the platinised glass, and to use this plate as the anode, the electrolytic liquid being retained as a capillary film between the edge of the metal plate and the glass. In the case of silver, a chemical reduction method due to Quincke was also employed, and as it was found impossible to obtain platinum prisms by electrolysis, they were formed by placing a strip of platinum foil perpendicular to the glass plate, and deflagrating it by a strong current, when it was deposited in a double wedge upon the glass. The prisms were carefully tested to ensure the surfaces being truly plane, by examining the reflection of a pair of fine crossed fibres, and the defective ones, which amounted to a large proportion of the whole, were rejected.

The following table gives the mean values obtained for the relative coefficients of refraction with respect to air, for red, white, and blue

light respectively. In the case of silver, the dispersion was too small to be determined.

Metal.	Mean values of μ.		
	Red.	White.	Blue.
Silver	—	0·27	—
Gold	0·38	0·58	1·00
Copper	0·45	0·65	0·95
Platinum	1·76	1·64	1·44
Iron	1·81	1·73	1·52
Nickel	2·17	2·01	1·85
Bismuth	2·61	2·26	2·13

The table shows that except in the cases of silver, which gives no appreciable dispersion, and of gold and copper, in which the dispersion is normal, all the metals examined produce anomalous dispersion.

Some experiments were made with prisms formed of iodide of silver formed by iodising a silver prism, and oxides of iron, nickel, bismuth, and copper, formed by heating prisms of the pure metal in the air. The results obtained are given below.

	Values of μ.		
	Red.	White.	Blue.
Iodide of silver	—	2·31	—
Oxide of iron	1·78	2·11	2·36
Oxide of nickel	2·18	2·23	2·39
Oxide of bismuth	—	1·91	—
Oxide of copper	2·63	2·84	3·18

Prisms formed of mixtures of platinum and gold with their oxides were also experimented on, but the values obtained varied considerably, owing doubtless to variations in the proportion of oxide present.

The results of the experiments appear to indicate that the velocities of light in different metals are in much the same proportion as the conductivities for electricity and heat. Thus taking the velocity in silver as 100, the velocities in the other metals are as below :—

Silver.	Gold.	Copper.	Platinum.	Iron.	Nickel.	Bismuth.
100	71	60	15·3	14·9	12·4	10·3

which correspond very closely with the relative electrical conductivities, except in the cases of copper and bismuth. The small value obtained for copper is probably due, as the author suggests, to

the unavoidable presence of oxide, which increases the value of μ, and, as is well known, the slightest traces of oxide largely diminish the electrical conductivity. In the case of bismuth, the authors point out that all the determinations of electrical conductivity have been made with crystalline specimens, for which the conductivity would probably be lower, whilst in the bismuth prisms used in these experiments no traces of crystalline structure was observable.

The author suggests that it would be of interest to further test the reality of the correspondence indicated, by endeavouring to determine whether μ increases with the temperature, in which case the velocity of light in the metal, like the electrical conductivity, would diminish with increase of temperature, and also whether magnetisation produces any effect on the value of μ, analogous to the different electrical conductivity in different directions, which is known to be produced in this manner. G. W. T.

Molecular Refraction of some Bromine-derivatives of Ethane and Ethylene, and the present Position of the Landolt-Brühl Theory. By R. Weegmann (*Zeit. physikal. Chem.*, **2**, 218—240, and 257—269).—These researches were undertaken with the object of testing the theories put forward by Landolt and Brühl of the connection between molecular refraction and chemical constitution, by the examination of a series of substances of simple, well-known constitution, but with high specific gravity and large refractive power, such as the bromine-derivatives of ethylene and ethane. The following table contains a list of the substances examined, together with the observed density d at 20° compared with that of water at 4°, and the refractive index n for the D line of the spectrum, and a temperature of 20°.

		d.	n.	$\frac{B}{d}$.
Ethylene chloride	$C_2H_4Cl_2$	1·25014	1·144387	0·35625
Ethylidene chloride	$C_2H_4Cl_2$	1·17503	1·416785	0·35623
Ethylene bromide	$C_2H_4Br_2$	2·17681	1·537888	0·34311
Ethylidene bromide	$C_2H_4Br_2$	2·05545	1·512767	0·34314
Acetylene tetrabromide	$C_2H_2Br_4$	2·96725	1·637951	0·33739
Acetylidene tetrabromide	$C_2H_2Br_4$	2·87482	1·627721	0·35515
Acetylene dibromide	$C_2H_2Br_2$	2·22889	1·543668	0·39949
Tribromethylene	C_2HBr_3	2·68762	1·599195	0·38881
Vinyl tribromide	$C_2H_3Br_3$	2·57896	1·589022	0·33868
Ethyl bromide	C_2H_5Br	1·45554	1·423865	0·34138
Aniline	C_6H_7N	1·02204	1·586321	1·34812
Benzene	C_6H_6	0·87907	1·501442	1·02886

The refractive index of each substance was also measured for the lines K, α, β, and γ, and for temperatures from below 10° to above 30°. The last column of the table contains the value of the second constant B in Cauchy's dispersion formula calculated from n_α and n_γ,

and may be taken as a measure of the dispersive power, divided, in order to free it from the influence of the density, by d. This gives to some extent a measure of the influence of constitution on the dispersion, and it is of interest that those substances containing a double bond give a nearly constant value of about 0·4, while the rest vary from 0·34 to 0·36. In other respects, Cauchy's formula was wholly unsatisfactory as an interpolation for refraction of different wave-lengths. The dispersion formula of Ketteler gave somewhat better results.

The specific refraction as given by both the formulæ $(n - 1)/d$ and $(n^2 - 1)/(n^2 + 2)d$ was calculated for each substance for the temperatures 10°, 20°, and 30°, and from this the molecular refraction. The specific refraction is not absolutely constant for the same substance, for the values given by the n formula decrease slightly with temperature, whilst those of the n^2 formula increase. This increase or decrease affects only the fourth decimal place for the 20° interval examined with either of the above formulæ; to three places of decimals, therefore, the constancy holds for this interval. The molecular refraction gives values on the whole confirmatory of the Landolt-Brühl theory. Differences which appear between the calculated values and those observed, may to some extent be explained by the want of constancy in the specific refraction, although probably chiefly due to the influence of dispersion. It is also found that of two isomeric substances the normal one always gives the smaller value for the specific refraction calculated by the n^2 formula. The two groups, acetylene dibromide, $C_2H_2Br_2$, ethylene or ethylidene bromide, $C_2H_4Br_2$, and tribromethylene, C_2HBr_3, vinyl tribromide, $C_2H_3Br_3$, form exceptions to the rule that substances containing a double bond have a specific refraction greater than, or at least equal to, that of compounds containing two additional hydrogen-atoms without the double bond. The atomic refraction of bromine calculated from the n_a values is for the n formula 15·38, and for the n^2 formula 8·83, as means of the experiments. H. C.

Luminescence of Pyrogallol. By P. Lenard and M. Wolf (*Ann. Phys. Chem.* [2], **34**, 918—925).—The authors, without knowing of the similar observations previously made by Eder (*Photogr. Mitt.*, No. 344), observed that when a photographic plate after development with an Eder potash developing solution, or with a soda developer after previous washing with a solution of alum, becomes distinctly luminous and remains so for a couple of minutes or so. The phenomenon appeared to them to be of sufficient interest to call for further investigation, and a number of experiments made with a view of determining the origin of the luminosity are described in the paper.

As the phenomenon was found not to be a case of true phosphorescence, they call it "*luminescence.*"

From their investigation, they arrived at the conclusion that the phenomenon is due to both pyrogallol and oxygen being condensed on the surface of the freshly precipitated alumina, resulting in rapid oxidation.

In general the luminosity was not observed when sodium sulphide was not present, but they found that a feeble luminescence could even then be produced by saturating potassium pyrogallate with pure oxygen and then adding a solution of alum. From this and other experiments they conclude, that the part played by the sodium sulphide is to delay the oxidation until the pyrogallol and oxygen have been brought into close contact in the pores of the precipitated alumina. G. W. T.

Fluorescence of Ferruginous Calcium Oxide. By L. DE BOISBAUDRAN (*Compt. rend.*, **106**, 1708—1710).—If calcium carbonate is mixed with a small quantity of ferric nitrate and strongly heated, the calcium oxide obtained shows a green fluorescence, which is sometimes very intense, but gives no definite spectrum. With only a few tenths of a per cent. of ferric oxide, the green fluorescence is well developed, and is less blue than that obtained in presence of copper. As the proportion of ferric oxide is reduced, the fluorescence gradually changes to that of pure calcium oxide. Similar results were obtained with ferric nitrate which had been purified by means of hydrogen sulphide. There can be no doubt that this green fluorescence is really due to iron and not to copper. C. H. B.

Degree of Oxidation of Chromium and Manganese in Fluorescent Mixtures. By L. DE BOISBAUDRAN (*Compt. rend.*, **106**, 1781—1784).—The green fluorescence shown by calcium oxide containing chromium is replaced by a white fluorescence if the mixture is strongly heated in a current of hydrogen. It is not due either to copper or to iron, and the fluorescence of calcium oxide containing lead is pale-yellow. All the specimens of calcium oxide which show this green fluorescence contain more or less chromium in the form of chromate. C. H. B.

Photochromatic Properties of Silver Chloride. By G. STAATS (*Ber.*, **21**, 2199—2200).—The author endeavoured to isolate the coloured substances formed on silver plates when the latter are treated with ferric chloride covered with variously coloured glasses, and exposed to sunlight (compare Abstr., 1887, 1671). Carey Lea's method (this vol., p. 1) was employed, but photo-red was obtained in all cases. Photo-red is produced when a silver plate of any colour is warmed. As soon as the superficial layer of the colour is treated with solvents, the colour is destroyed, but when very thin plates of chemically pure silver are treated with a solution of ferric chloride on one side only, then exposed in the usual manner until the red and violet colours appear, and finally dissolved in nitric acid, both the red and violet separate unchanged in thin plates, which are generally bounded by straight lines.

The separated silver chloride which has not been coloured is built up of small yellow plates. F. S. K.

Constant Battery with a Negative Electrode of Carbon. By FABINGI and FARKAS (*Compt. rend.*, **106**, 1597—1598).—In this

battery the negative electrode consists of carbon and the positive electrode of platinum, and there are two electrolytes, namely, fused sodium or potassium hydroxide, and the product of the action of fused sodium hydroxide on platinum, $Na_2O,PtO_2 + 6H_2O$, or $2Pt(OH)_4ONa + 3H_2O$. The latter compound covers the internal surface of a small platinum capsule, which is then filled with sodium hydroxide, previously fused in a silver dish. A small gas flame serves as a source of heat, and an electrode of gas carbon is immersed in the fused sodium hydroxide, the whole apparatus being protected from draughts, &c., by a screen. When the circuit is completed the electromotive force quickly reaches a certain value, and the action then becomes stationary. The electromotive force varies in the same direction as the temperature in the neighbourhood of the carbon, and in the opposite direction in the neighbourhood of the platinum, and, other conditions being equal, the intensity of the latter is greater than that of the former. When the two temperatures are equal, the electromotive force varies in the same direction as the temperature. When the variations in the height of the flame are small, and the loss of fused sodium hydroxide does not exceed a certain limit, the variations in the electromotive force are inappreciable. Sudden and considerable variations in the external resistance of the circuit produce only very brief temporary variations in the difference of potential between the two electrodes. C. H. B.

Use of Magnesium in Primary Batteries. By C. Heim (*Electrotechnische Zeitschrift*, **8**, 472—476, and 517—524).—These investigations were undertaken with a view to utilise magnesium practically for primary batteries for laboratory use, as recently invented methods of manufacture enable it to be produced at a much lower cost than formerly.

The temperature of observation was in every case between 17° and 21°. The following solutions were used throughout the experiments; the specific gravities being determined at a temperature of 15°. Dilute sulphuric acid, 1·026; magnesium sulphate, 1·223; magnesium chloride, 1·170; sodium chloride, 1·155; copper sulphate, 1·187.

The values of the E.M.F. given by a zinc-magnesium couple in a single fluid were in volts:—

In sulphuric acid, 0·876; in magnesium sulphate, 0·784; in magnesium chloride, 0·769; in sodium chloride, 0·616.

The values of the E.M.F. obtained by replacing the zinc in a Daniell, Bunsen, dichromate, and Leclanché cell respectively are given opposite, the E.M.F. of the normal cell being given for comparison.

The dichromate solution consisted of 92 parts potassium dichromate, 172 parts sulphuric acid, and 900 parts water, as used by Bunsen. At 15° its specific gravity was 1·140. The specific gravity of the concentrated solution at the same temperature was 1·261.

Of the different cells experimented with, the only ones found to be of practical value were the modifications of the Bunsen and Leclanché cells, in which magnesium in a solution of its sulphate or chloride was

Name of cell.	Anode.	Liquid or mixture surrounding anode.	Kathode.	Liquid surrounding kathode.	E.M.F. in volts.
Daniell	Copper ..	Copper sulphate	Zinc	Dilute sulphuric acid	1·183
	,,	,,	Magnesium .	,,	2·033
	,,	,,	,,	Magnesium sulphate	1·930
Bunsen	Arc lamp carbon	Nitric acid containing 68 p. c. of acid	Zinc	Dilute sulphuric acid	1·991
	,,	,,	Magnesium .	,,	2·888
	,,	,,	,,	Magnesium sulphate	2·863
	,,	,,	,,	Magnesium chloride	2·910
	,,	,,	,,	Sodium chloride	2·709
Dichromate .	Arc lamp carbon	Dichromate solution	Zinc	Dichromate solution	2·000
	,,	,,	Magnesium .	,,	2·952
	,,	,,	Zinc	Dilute sulphuric acid	2·110
	,,	,,	Magnesium .	,,	2·980
	,,	,,	,,	Magnesium sulphate	2·900
	,,	,,	,,	Magnesium chloride	2·970
	,,	Concentrated dichromate solution	,,	Dilute sulphuric acid	3·042
	,,	,,	,,	Magnesium sulphate	2·971
	,,	,,	,,	Magnesium chloride	3·046
Leclanché ..	Carbon ..	Mixture of coke and manganese dioxide in small grains	Zinc	Ammonium chloride	1·587
	,,	,,	Magnesium .	,,	2·219
	,,	,,	,,	Magnesium sulphate	2·334
	,,	,,	,,	Magnesium chloride	2·364
	,,	,,	,,	Sodium chloride	2·151

substituted for zinc in dilute sulphuric acid. Of these two solutions, the chloride is preferable, chiefly on account of its specific conductivity being about three times that of the sulphate. In the Bunsen cell, the initial E.M.F. is slightly higher with the chloride than with the sulphate, but it does not remain quite so constant. In the Leclanché cell, the initial E.M.F. is again slightly higher with the chloride,

but it also remains much more constant than when the sulphate is used.

The tables given below show the relative behaviour of these and the normal cells of equal dimensions. T is the time in hours from the closing of the circuit, E the E.M.F. in volts, R the internal resistance in ohms, and C the current in ampères through an external resistance adjusted to give as nearly as possible an initial current of 1 ampère.

Normal and Modified Bunsen Cells.

T.	Zinc in dilute sulphuric acid.			Magnesium in magnesium sulphate.			Magnesium in magnesium chloride.		
	E.	R.	C.	E.	R.	C.	E.	R.	C.
0	1·931	0·35	1·081	2·809	0·55	1·015	2·826	0·41	1·028
0·5	1·853	0·18	1·162	2·725	0·46	1·035	2·705	0·28	1·040
1	1·841	0·16	1·131	2·701	0·45	1·030	2·647	0·23	1·030
2	1·837	0·15	1·117	2·661	0·54	0·982	2·589	0·21	1·014
3	1·827	0·15	1·147	2·600	0·54	0·960	2·587	0·26	0·989
4	1·828	0·16	1·130	2·565	0·53	0·961	2·570	0·29	0·976
6	1·817	0·18	1·113	2·532	0·50	0·952	2·522	0·35	0·934
7	1·808	0·19	1·095	2·519	0·52	0·944	2·497	0·37	0·922
8	1·803	0·21	1·092	2·492	0·49	0·940	2·487	0·42	0·894
9	1·798	0·23	1·050	2·488	0·50	0·934	2·470	0·44	0·891

Normal and Modified Leclanché Cells.

T.	Zinc in ammonium chloride.			Magnesium in magnesium sulphate.			Magnesium in magnesium chloride.		
	E.	R.	C.	E.	R.	C.	E.	R.	C.
0	1·646	2·4	0·1019	2·300	3·8	0·1013	2·306	2·5	0·1009
0·5	1·451	1·1	0·0977	2·199	3·2	0·0991	2·222	2·2	0·0988
1	1·428	1·1	0·0960	2·152	3·1	0·0976	2·180	2·2	0·0970
2	1·396	1·1	0·0940	2·150	3·5	0·0954	2·157	2·2	0·0960
3	1·368	0·95	0·0930	2·072	3·1	0·0939	2·132	2·2	0·0951
4	1·355	0·91	0·0924	2·047	3·1	0·0925	2·120	2·2	0·0945
6	1·333	0·85	0·0910	2·014	3·2	0·0906	2·098	2·3	0·0933
7	1·323	0·84	0·0907	2·006	3·4	0·0897	2·090	2·2	0·0930
8	1·306	0·72	0·0900	1·994	3·4	0·0891	2·090	2·2	0·0927
9	1·309	0·81	0·0895	1·983	3·4	0·0886	2·074	2·2	0·0927

These modifications of the Bunsen cell are suitable for use where a strong current is often required for a moderate time, and the modifications of the Leclanché where a high and constant E.M.F. is required to give a small current through a large resistance. G. W. T.

Determination of the Potential Differences between Mercury and Electrolytes. By R. Blondlot and E. Bichat (*Zeit. physikal. Chem.*, **2**, 98—99).—Reply to objections raised by Ostwald against the accuracy of the authors' method of experimenting, in which, while admitting that the method devised by the latter is easier of performance and leads to better results, their own is defended in point of accuracy. H. C.

Electrolysis with Alternating Currents. By G. Maneuvrier and J. Chappuis (*Compt. rend.*, **106**, 1719—1722; and **107**, 37—40).—When an alternating current is passed through an ordinary voltameter containing acidulated water, there is usually no appreciable decomposition. If, however, electrodes of very slender platinum wire are used in place of platinum plates, there is an immediate and abundant evolution of gas. For a given intensity of current there is a particular area of surface of electrode from which no gas is evolved, or in other words, with which no electrolysis takes place, but if the surface is gradually reduced by reducing either the length or breadth of the electrode, evolution of gas begins, and is greater the greater the reduction of surface. Conversely, for an electrode with a given surface there is a particular intensity of current below which there is no evolution of gas. Above this limit electrolysis takes place, and increases with the intensity of the current. The gas evolved has the same composition in all cases, but the proportion of oxygen is less than one-third of the total volume. Ozone is produced in considerable quantity at both electrodes, notwithstanding the presence of hydrogen. The proportion varies with the intensity of the current, and also with the form of the electrodes.

The simultaneous presence of ozone and hydrogen at the electrodes points to their reconversion into water, and with platinum electrodes a determining cause is to be found in the condensing power of the surface of the metal, the condensing action being proportional to the area of surface.

With copper electrodes there is no evolution of gas, even when the intensity of current is sufficient to produce electrolysis with platinum electrodes, but the surface of the copper becomes covered with reduced copper, and hence it is evident that the ozone converts the copper into oxide, and the latter is reduced by the hydrogen. If copper wire is employed for the electrodes there is evolution of gas, but the action soon ceases, since the wire disintegrates and falls to the bottom of the voltameter in the form of reduced copper.

Mercury electrodes with a large surface cause a slight evolution of hydrogen, and the surface of the metal becomes covered with mercurous sulphate. Under similar conditions a continuous current gives mercuric sulphate.

It is evident from these observations that the primary and secondary reactions with alternating currents are very different from those obtained with direct currents.

When an alternating current of 2·5 ampères is passed through a voltameter with platinum electrodes containing a solution of copper sulphate, there is considerable development of heat, but no electro-

lysis. If, however, the electrodes are reduced to 0·1 mm. diameter and 20 mm. in length, gas is evolved, and copper is deposited. The same phenomena are observed with electrodes of copper, but the latter are gradually converted into spongy reduced copper, the metal being alternately oxidised and reduced.

In electrolysis with alternating currents, a condition of equilibrium is always possible between the velocity of electrolytic decomposition and the velocity of recombination of the constituents of the electrolytes. The chief cause tending to increase the rate of decomposition is the intensity of the current. The electrodes may also exert considerable influence either by taking part in the chemical change or by reason of their physical properties. Other conditions being equal, the frequency of alternation of the current is an important factor. If the alternations are comparatively slow, there is sufficient time for the products of decomposition to diffuse away from the immediate neighbourhood of the electrodes, and therefore out of the sphere of possible recombination.

By means of a dynamo with separate excitation, the authors were able to vary the rate of alternation of a current whilst keeping its intensity constant. In one experiment the intensity of the current was adjusted so that with 2000 alternations per minute there was no electrolysis of acidified water, but if this was reduced to 1500, there was an abundant evolution of gas. In another case there was a regular evolution of gas with 2000 alternations per minute, but when this was increased to 2600, decomposition ceased. Similar effects are produced by varying the superficial area of the electrodes.

C. H. B.

Alternate Current Electrolysis. By W. E. Ayrton and J. Perry (*Electrician*, **21**, 299—300).—This paper contains an account of some detailed experiments made under the authors' direction by Messrs. Raine, Lamb, Smith, and Priest, and was published in its present fragmentary form in connection with the paper recently communicated to the French Academy by Maneuvrier and Chappuis (preceding Abstract).

The authors state that with an ordinary voltameter no evolution of gas is as a general rule observed with alternate currents. In the Central Institution experiments, on the contrary, alternate currents passed through an ordinary Hoffmann voltameter containing a 33 per cent. solution of sulphuric acid, always caused an evolution of gas. The gas comes off, as far as can be seen, from the wires instead of from the plates, as with direct currents, and the liquid is found to get rapidly hot, showing that there must be a good deal of decomposition and recomposition of the gases. An alternate current of 3 ampères passing through a Varley resistance, made of flexible carbon cloth, in circuit with the voltameter, was found to heat the former much more than a direct current of the same strength. This was probably due to the rapid decomposition and recomposition of the moisture on the cloth.

According to Maneuvrier and Chappuis, a current density of at least 3 ampères per sq. cm. is requisite to produce visible decomposition, whilst according to these experiments a current-density of less

than one-third of this, counting only one surface of each plate, or one-sixth, counting both surfaces, is required, unless practically the whole of the current is to be supposed to pass from wire to wire, and none to leave the plates.

The rate of alternation in the experiments was about 10,080 per minute.

When the speed of the dynamo was increased, thereby increasing the rate of alternation, the decomposition became much less, although the current at the same time increased from 3 to $4\frac{1}{2}$ ampères. The speed was then lowered until the current was again 3 ampères, but the decomposition was still much less than in the first case, while gas was evolved about twice as fast at one electrode as at the other, the former consisting of nearly pure hydrogen, and the latter of an explosive mixture. When the same voltameter was used again, gas came off freely from one electrode and not from the other, and when the dynamo terminals were reversed, the gas continued to come off only from the same electrode as before. When a direct current was used, gas came off freely from both electrodes, but on passing an alternate current again, the same thing occurred just as before.

In some further experiments with about 10,000 alternations per minute, it was found that one electrode, say P, was better able to produce visible decomposition than the other, Q. The superiority of P over Q was gradually diminished by sending a direct polarising current from Q to P through the voltameter, and finally the superiority was reversed. When the direct polarisation current was reversed P gradually regained its superiority. With an alternate current of $2\frac{1}{2}$ ampères, and a direct polarising current of 6 ampères, it was found possible to cause the former to produce rapid decomposition at either of the electrodes at will, and no visible decomposition at the other electrode, by previously sending for some time the 6 ampère current through the liquid from that electrode at which subsequent decomposition by the alternate current was desired.

It seems, therefore, that a deposition of hydrogen on a platinum electrode hinders, and may even stop altogether all visible decomposition by an alternate current, while a deposition of oxygen on the other electrode does not hinder, but may even favour visible decomposition.

Further it appears that although the direct polarising current may be strong enough to produce rapid decomposition, the gases so produced do not affect in any marked way the subsequent relative decomposition produced at the two electrodes by an alternate current, when the alternate current exceeds some particular strength relatively to that of the previous direct polarising current.

G. W. T.

Electrolysis of Solutions of Potassium Hydroxide. By G. Berson and A. Destrem (*Compt. rend.*, **106**, 1794—1797).—When a solution of potassium hydroxide is electrolysed with platinum electrodes, the volume of the oxygen evolved is always less than half that of the hydrogen, and the liquid near the positive electrode gives the reactions for hydrogen peroxide. With a positive electrode of

copper, a 50 per cent. solution of potash, and a current of 0·06 ampère, the extremity of the copper electrode blackens as soon as the circuit is closed, and a blue cupropotassium solution falls from it to the bottom of the voltameter. This black coating gradually spreads over the surface of the electrode, and as soon as it is completely covered, there is an evolution of gas, and the resistance of the electrolyte increases by about 50 per cent. The black layer of oxide gradually dissolves, forming a cupropotassium solution, and the perfectly clean copper electrode then behaves exactly like platinum. If the circuit is broken, and then quickly closed again, the electrode continues to behave like platinum, but if there is a considerable interval of time before the circuit is again completed, the black layer is again formed on the copper, and the series of phenomena is repeated. It would seem, therefore, that the insolubility of the copper is due to the formation of a layer of gas on the surface of the electrode.

For the same current and the same liquid, the time which elapses before the electrode is covered with oxide, and the evolution of oxygen begins, is proportional to the surface of the electrode, a fact which is readily illustrated by inclosing in the same circuit two voltameters, in one of which the positive electrode is a copper plate, and in the other a copper wire. For a given solution and a given electrode, the time is shorter the greater the intensity of the current. With weak currents, the layer of oxide does not dissolve. The time is also shorter the weaker the potash solution. C. H. B.

Determination of the Size of the Molecules of Salts from the Conductivity of their Aqueous Solutions. By P. Walden (*Zeit. physikal. Chem.*, **2**, 49—77).—In continuation of the author's researches on the above (this vol., p. 891), it appears from the examination of the sodium salts of arsenious acid, that a tribasic acid does not exist, salts of the form $As(OMe')_3$ being basic salts, and that arsenious acid in solution behaves as a dimeta-acid, having the molecular formula $(AsO{\cdot}OH)_2$. It is shown that mono- and bi-sodium arsenates, both of which exist as such in solution, are converted respectively by heating wholly into the meta- and pyro-arsenates. Trisodium arsenate is unstable in solution, and decomposes with rising dilution. In arsenic acid, as in phosphoric acid, the three hydrogen atoms are of unequal value. The monosodium salt is strongly acid, the bisodium, which appears to be the normal salt, is neutral, and the trisodium is basic.

Hydrofluoric acid does not exist as bibasic acid, H_2F_2, in dilute solution, but as the simple molecule HF, and the acid fluorides are decomposed into normal salt and free acid. Chloric, bromic, and also iodic acid are monobasic, the so-called acid iodates not existing in solution. Measurements with periodates show that of these only the mono-salt is stable, both sodium periodate and perchlorate being salts of monobasic acids. Increasing the amount of alkali results in the formation of unstable and basic salts.

The salts of the bibasic chromic acid are less stable than the isomorphous sulphates, but are better conductors. Acid salts of the form $R'HCrO_4$ do not exist. Di- and tri-chromates are unstable, and

do not exist undecomposed in solution. Platinocyanic and platinochloric acids are bibasic, the salts being atomic and not molecular compounds. Salts of the form R'HPtCy$_4$ do not exist in solution, but separate into neutral salt and free acid. Chromicyanic is analogous to ferricyanic acid and tribasic. H. C.

Chromic Acid. By W. Ostwald (*Zeit. physikal. Chem.*, **2**, 78—80).—The results of Walden's researches on chromic acid (preceding Abstract) show that it cannot be included in the list of ordinary mono- or bi-basic acids, and this together with the high conductivity of its solutions, which appears from the very slight increase with rising dilution to be near its maximum, and general considerations of its behaviour, lead the author to conclude that in aqueous solution the acid exists not as H_2CrO_4 but as $H_2Cr_2O_7$. The following is brought forward in support of this.

From the recent investigations of the molecular reduction of the freezing point of water by various substances, it appears that the freezing point of a solution which contains in a kilogram 1 gram-molecule of any compound should be $-1{\cdot}85°$; or, in the case of an acid, if complete dissociation of the compound into its ions takes place, it is double this if the acid is monobasic and treble if it is bibasic. If instead of this equivalent normal solutions be taken, in the latter case the number for the bibasic acid would have to be halved, and thus give a value three-quarters of that for the monobasic acid. The following freezing points were observed for equivalent normal solutions: $C_2H_4O_2$ $-1{\cdot}92°$; HNO_3 $-3{\cdot}70°$; H_2SO_4 $-2{\cdot}00°$; H_2CrO_4 $-1{\cdot}34°$. Acetic acid not being dissociated gives the normal value; nitric acid in which dissociation is nearly complete about double this; and sulphuric acid which is dissociated about 0·6; and the solution of which contains therefore $\frac{1}{2}(0{\cdot}4 + 3 \times 0{\cdot}6) = 1{\cdot}10$ mol., should and does freeze at $-2{\cdot}00°$. Chromic acid, if H_2CrO_4, and in accordance with the conductivity dissociated one-half, should have the freezing point of a normal solution $-1{\cdot}85°$, but if $H_2Cr_2O_7$, it follows from the conductivity that this would be completely dissociated, and that the freezing point of the solution should be $-1{\cdot}85 \times \frac{1}{4} \times 3 = -1{\cdot}39°$. The number actually obtained is 1·34°. H. C.

Apparent Manifestation of Chemical as Mechanical Attraction. By J. W. Langley (*Zeit. physikal. Chem.*, **2**, 83—91).—An arrangement was devised by which any change of weight in one of the electrodes of an electrolytic cell could be ascertained, and the current changed in direction at the will of the observer, so that this electrode might be converted into either positive or negative pole. In the first series of experiments, the electrodes were of copper. On immersing them in saturated copper sulphate containing only traces of free sulphuric acid and passing the current, in contradiction to the course of the experiment which requires the transference of copper from the positive to the negative pole, the positive pole was observed to gain in weight and the negative pole to become lighter, this change lasting for from 20 seconds to one minute, and then a gradual reversal set in and the normal action took place. From this it appears that

this change may be divided into two epochs, the first a variable and reversible one, and the second permanent.

A similar peculiarity was noted for solutions of the following salts, the electrodes being in each case the same metal as that of the salt: $Cu(NO_3)_2$, $ZnSO_4$, $ZnCl_2$, $ZnBr_2$, ZnI_2. The acetates of copper, zinc, and lead act normally, and from this and slight differences observed with salts of the same metal, it appears that the change depends primarily on the character of the negative radicle. The acetates probably only form an apparent exception, their behaviour being due to the comparatively feeble acidity of acetic acid, as no change in weight whatever occurs in either electrode until after the first 10 or 20 seconds of the current, the permanent change then gradually setting in. Experiments were then made with free acids which show more clearly that the variable epoch depends in the first degree on the nature of the negative radicle. The metals zinc, copper, mercury, and platinum all give the variable increment in weight at the positive pole, the strength of the acid solution making little or no difference.

On weighing the electrodes before and after electrolysis, it was found that both during the variable and permanent epoch the positive pole was losing in weight. The phenomenon of the variable epoch must therefore be caused by some change in the solution surrounding the electrode and not in the electrode itself. By further experiments it is shown that a concentration of the negative radicle takes place round the positive pole. This concentration, due to the affinity of the metal of the electrode for the negative radicle, is, according to hypothesis, the result of a linear attraction extending to a distance far exceeding the radius of one molecule and of an elective kind. While this attraction is in force, that is, during the passage of the current, a layer of molecules of the negative radicle is produced on the positive electrode, thus causing its increase in weight.

H. C.

Expansion, Compressibility, and Specific Heat of Solutions of the Chlorides of Potassium and Calcium. By J. Drecker (*Ann. Phys. Chem.* [2], **34**, 952—970).—It is found that in general the specific heat of a solution or mixture differs from the mean calculated from the specific heats of the constituents. This might be due to a difference in the internal work of expansion in the mixture and in the separate constituents, but if this were the only cause the observed specific heat at constant volume would agree with the value calculated from the specific heats of the constituents.

The author, in his previous investigations (*Ann. Phys. Chem.* [2], **20**, 870), found that for a large number of liquids the observed and calculated specific heats at constant volume agreed more closely than those at constant pressure.

The whole of the observed differences, however, cannot be explained by differences in the internal work of expansion.

The present paper contains an account of similar experiments made with solutions of the chlorides of potassium and of calcium of different concentrations.

The author finds that the compressibility of these solutions is less

than that of water, but the diminution is not proportional to the quantity of salt in solution. Up to a certain concentration, the compressibility increases with the temperature, as in the case of water, and afterwards it diminishes as in the case of alcohol, ether, chloroform, and other liquids. The concentration for which the compressibility is independent of the temperature is about 16 per cent. for potassium chloride and 20 per cent. for calcium chloride.

He confirms Braun's result (Abstr., 1887, 436), that the strength of a saturated solution of a salt depends on the temperature, but finds that Röntgen and Schneider's equation (*Ann. Phys. Chem.* [2], **29**, 165) for determining the compressibility of a solid salt from that of its solution is only a very rough approximation.

The specific heats of the solutions, both at constant pressure and constant temperature, were found to be less than those of water when the solutions were dilute, and the differences increased with the concentration up to a certain amount, when they reached a maximum, and decreased with further concentration until, with sufficiently concentrated solutions, the specific heat was greater than that of water.

The differences between the observed specific heats and their calculated values were greater at constant volume than at constant pressure, showing that the internal work of expansion of the solutions is greater than that of the constituents, so that the behaviour of the solutions is in this respect exactly the opposite to that of the mixtures of liquids formerly investigated. G. W. T.

Heat of Formation of Mercury Compounds. By J. Thomsen (*Zeit. physikal. Chem.*, **2**, 21—22).—Ostwald has pointed out that the reduction of mercurous nitrate by sulphurous acid, resulting in the separation of metallic mercury, the reaction by which the author formerly determined the heat of formation of mercurous oxide, does not take place with the assumed regularity, and that consequently the thermal data based on the above determination contain a constant error from this source. In order to obtain some idea of the magnitude of this error, the author has determined the heat evolved when mercurous nitrate is decomposed by a solution of iodine in potassium iodide resulting in the formation of mercury potassium iodide, and also when it is decomposed by potassium iodide alone, resulting in the separation of one-half of the mercury in the metallic state, and the conversion of the other half into mercury potassium iodide, the difference between these two reactions conducted under similar conditions being the heat evolved by the reaction $HgI_2,nKIAq$. Thus determined and calculated for one molecule of mercurous nitrate this quantity is 29090 cal., and as the former determination by means of the reduction experiment gave 37760 cal., the constant error of this latter method would appear to be 8670 cal. On this assumption, the corrected heats of formation of the following compounds would be:—

Hg,Cl_2	54490 cal.	Hg_2,Cl_2	65210 cal.
Hg,Br_2	41880 ,,	Hg_2,Br_2	50950 ,,
Hg,I_2	25640 ,,	Hg_2,I_2	31100 ,,
Hg,O	22000 ,,	Hg_2,O	24860 ,,
Hg,S	8220 ,,	Hg,Cy_2	10280 ,,

H. C.

Heat of Formation of Mercury Compounds. By W. NERNST (*Zeit. physikal. Chem.*, **2**, 23—28).—The author has determined directly the heat of formation of mercuric bromide from liquid mercury at about 15°, and finds it to be 40460 cal. per gram-molecule, the number calculated by Thomsen from the reduction of mercurous nitrate by sulphurous acid (preceding Abstract) being 50600 cal. Taking the finely divided mercury obtained in the latter reaction and substituting it for the liquid mercury before used, a number about 12 per cent. higher was in fact obtained, but on examination, after repeated washings, the mercury was found still to contain nitric and sulphuric acids. This, together with the fact that in the finely divided state the mercury is already in a condition which favours the reaction, will perhaps serve to explain the discrepancy.

By means of the number obtained for mercuric bromide, the following heats of formation have been recalculated from Thomsen's numbers:—

Hg,Cl_2	53200 cal.	Hg_2,Cl_2	62600 cal.
Hg,I_2	24300 ,,	Hg_2,I_2	28400 ,,
Hg,O	20700 ,,	Hg_2,O	22200 ,,

The heat of formation of mercuric from mercurous bromide was directly observed to be 16000 cal., and this would give for the heat of formation of mercurous bromide 24500 cal. By means of these new numbers the calculated E.M.F. of various galvanic elements containing mercury as one pole, is brought into agreement with the observed values, which has hitherto not been the case, using the numbers obtained from Thomsen's determination.

The heat of formation of cadmium bromide in aqueous solution was also determined and found to be 76300 cal., Thomsen's number, obtained by totally different method, being 75600 cal. H. C.

Heat of Formation of Salts of Phenylenediamine. By L. VIGNON (*Compt. rend.*, **106**, 1671—1674).—Paraphenylenediamine crystallises from a boiling saturated aqueous solution in transparent, white, monoclinic lamellæ which contain 2 mols. H_2O, melt at 80°, effloresce in a vacuum, and become rose-coloured when exposed to air. The normal sulphate, $C_6H_4(NH_2)_2,H_2SO_4$, forms micaceous plates and is only slightly soluble in water, the solution being acid to litmus. The oxalate also forms anhydrous crystals which do not melt and are very slightly soluble in water, the solution being acid to litmus.

Heat of solution of anhydrous paraphenylenediamine −3·8 Cal., of the hydrate −7·4 Cal., whence

$$C_6H_4(NH_2)_2 + 2H_2O = C_6H_4(NH_2)_2{\cdot}2H_2O$$

develops +3·6 Cal.

The other values obtained were as follows:—

$C_6H_4(NH_2)_2$ diss. + HCl dil.	develops	+8·8 Cal.
$C_6H_4(NH_2)_2$ diss. + 2HCl dil.	,,	+14·7 ,,

$C_6H_4(NH_2)_2$ diss. + $\frac{1}{2}H_2SO_4$ dil.	develops	+9·6 Cal.
$C_6H_4(NH_2)_2$ diss. + H_2SO_4 dil.	„	+19·2* „
$C_6H_4(NH_2)_2$ diss. + AcOH dil.	„	+7·2 „
$C_6H_4(NH_2)_2$ diss. + 2AcOH dil.	„	+8·4 „
$C_6H_4(NH_2)_2$ diss. + $\frac{1}{2}H_2CO_3$ dil.	„	+8·8 „
$C_6H_4(NH_2)_2$ diss. + H_2CO_3 dil.	„	+22·2 „

It is evident that the neutralisation of one of the basic functions of paraphenylenediamine reduces the intensity of the remaining function. So far at least as the first function is concerned, paraphenylenediamine is a more energetic base than aniline. C. H. B.

Heat of Neutralisation of Aromatic Amines. By L. Vignon (*Compt. rend.*, **106**, 1722—1724).—The following results were obtained:—

	Hydrochloric acid.	Sulphuric acid.	Acetic acid.
Aniline, 1st equivalent	7·38	8·75	3·85
„ 2nd „	0·92	0·71	2·14
Methylaniline, 1st equivalent	6·91	8·06	—
„ 2nd „	1·31	0·92	—
Dimethylaniline, 1st equivalent.....	6·81	7·60	—
Dimethylaniline, 2nd equivalent.....	1·02	0·71	—

The quantity of heat developed by the successive action of one and two equivalents of acid on the amines affords a measure of the heats of formation of the different salts and also a measure of the degree to which the salts are dissociated in dilute solution. With aniline and acetic acid the action is far from complete. A comparison of the anilines with ammonia and the methylamines shows that the substitution of methyl for hydrogen in ammonia or in the NH_2 group in aniline has little effect on the energy of the basic function, whereas the substitution of phenyl for hydrogen in ammonia very considerably reduces the activity of this function. C. H. B.

Heats of Combustion of Organic Compounds. By F. Stohmann (*Zeit. physikal. Chem.*, **2**, 29—35; comp. Abstr., 1887, 878, 1011).—Contains the tabulated results of the author's published experiments on the above, giving the heat of combustion per gram and per gram-molecule of each substance, and the heat of formation calculated from this, using Berthelot's numbers, C = 94 Cal. and H_2 = 69 Cal. H. C.

Thermo-chemistry of Nitrogen-derivatives of Benzene. By P. Petit (*Compt. rend.*, **106**, 1668—1669).—The heats of combustion were determined by means of the calorimetric bomb, and are given in the following table, in which A is the heat of combustion per gram-molecule at constant volume, B the corresponding value under constant pressure, and C the heat of formation from the diamond, hydrogen, and nitrogen.

* Crystallised.

	A.	B.	C.
Azoxybenzene	1529·55	1530·6	−57·6 Cal.
Azobenzene	1555·0	1556·4	−83·4 „
Hydrazobenzene ..	1598·0	1599·6	−57·6 „
Phenylhydrazine ..	805·24	806·3	+33·7 „

The formation of azobenzene from aniline and nitrosobenzene, C_6H_7N liq. + C_6H_6 liq. + HNO_2 dil. = $C_{12}H_{10}N_2$ + $2H_2O$ liq., develops +42·3 Cal. The formation of phenylhydrazine from aniline and hydroxylamine, C_6H_7N liq. + NH_2OH dil. = $C_6H_8N_2$ + H_2O, develops +96·7 Cal.; the formation of phenylhydrazine hydrochloride, $C_6H_8N_2$ liq. + HCl dil. = $C_6H_8N_2$,HCl diss., develops +8·9 Cal.; the heat of dissolution of the hydrochloride is −5·96 Cal., and hence $C_6H_8N_2$ liq. + HCl gas = $C_6H_8N_2$·HCl solid, develops +32·2 Cal., a value which approaches the heat of formation of ammonium chloride by the corresponding reaction. C. H. B.

Disintegration of Glowing Platinum. By H. Kayser (*Ann. Phys. Chem.* [2], **34**, 607—608).—In some determinations of the tension coefficient of air as a function of the pressure, which the author had been making, he passed the air through a glass tube containing platinum wires heated to redness in order to remove traces of hydrocarbons. He found that the result was to considerably increase the tension coefficient, which however returned to its normal value when the air was allowed to stand for some time after passing over the heated platinum.

He concluded that the phenomenon was due to the presence of disintegrated particles of platinum, and the conclusion was confirmed by the fact that the tension coefficient immediately returned to its normal value when the air was filtered by being forced under pressure through very tightly packed cotton-wool. G. W. T.

Viscosity of Gases at High Temperatures, and the Pyrometric Use of the Principle of Viscosity. By C. Barus (*Amer. J. Sci.*, **35**, 407—410).—By passing gases under known conditions through capillary platinum tubes maintained at known temperatures varying from 5° to 1400°, the author has obtained a series of data for determining the relation existing between the viscosity of a gas and its temperature.

The results obtained show that for the range of temperature within which the observations were made, the mean increase in the viscosity of a gas is proportional to T^1, where T is the absolute temperature, and taking into account the relations established by Clausius and Maxwell, it is found that the mean free path of a molecule of a perfect gas varies directly as the sixth root of the absolute temperature, a result of considerable importance, as the condition that the length of the mean free path should be independent of the temperature is usually regarded as a criterion of a perfect gas.

If this law of the sixth root is assumed as the criterion of a perfect gas, namely, one in which no transient mechanically cohering molecular aggregates are formed, it follows that the mean free path must be proportional to the cube root of the velocity of the mean square.

If the exact law of thermal variation of the viscosity of a gas were known, Poiseuille-Meyer's equation applied to transpiration data would give us a means of measuring temperature absolutely over a wider range, and with an accuracy hitherto unattainable. The author suggests that the method may be advantageously applied to the study of the phenomena of gaseous dissociation, of phenomena at temperatures in the neighbourhood of the critical temperature, and for the determination of vapour-pressure at high temperatures.

G. W. T.

Change of Volume in Gases on Mixture: Is the Pressure of a Saturated Vapour Independent of the Presence of other Gases? By F. Braun (*Ann. Phys. Chem.* [2], **34**, 943—952).—The question whether the presence of a saturated vapour is the same in a vacuum as in the presence of a gas is still a disputed one. Regnault attributed the differences observed by him to secondary causes, such as the deposition of condensed vapour on the walls of the containing vessel.

The author describes a series of careful experiments on sulphurous anhydride, from the results of which he arrives at the conclusion that the pressure of this vapour at the temperature of condensation is diminished by at least 3·9 mm. of mercury by the addition of an equal volume of carbonic anhydride at the same pressure and temperature, and by 1·3 mm. by the addition of an equal volume of nitrogen under similar circumstances. He infers in general that the pressure of a saturated vapour is not the same in the presence of a gas as in a vacuum, even when all disturbing causes are absent.

The question is of importance in relation to the dissociation of gases, and up to the present it has not been found possible to decide it on theoretical grounds.

G. W. T.

Determination of the Specific Weight and Vapour-pressure of Mixtures of Sulphurous and Carbonic Anhydrides.—By R. Pictet (*Ann. Phys. Chem.*, [2], **34**, 734—736).—The author states that in a work in the press on "Machines for Freezing by Compression," he has criticised at length the results given in A. Blumcke's paper (this vol., p. 775), and in the meantime he calls attention to the following points:—

(1.) In his earlier work on the subject, he has pointed out that no satisfactory determination has yet been made of the chemical constitution of a liquid boiling at −19°, and that the formula SCO_4, analogous to SO_3H_2, must only be looked on as a label adopted for convenience.

(2.) The principal result of his investigations consisted in the establishment of the fact that the mobile liquid boiling at −19° has at 30° a vapour-pressure of only 4·5 atmospheres instead of 8, as theory would lead us to expect.

(3.) Blumcke found this vapour-pressure to be 4·6 instead of 4·5, a smaller difference than that existing between different determinations of Regnault's.

(4.) Blumcke's results were obtained by means of Cailletet's apparatus, and during the compression and liquefaction of the gas

its volume was reduced to 1/300 or 1/400 of its original value, so that the slightest trace of a foreign gas would be sufficient to introduce an error of two or three atmospheres, and he considers that this is the source of the errors which Blumcke thought he had found.

(5.) Blumcke's own determinations of the vapour-pressure of sulphurous acid differed by from 0·18 to 0·92 of an atmosphere from those obtained by Regnault, and he thinks that Blumcke has not given sufficient weight to these deviations. G. W. T.

Determination of Vapour-pressure from the Rate of Evaporation. By W. Müller-Erzbach (*Ann. Phys. Chem.* [2], **34**, 1047—1048).—The author observes that G. Tammann (this vol., p. 403) had made some determinations of the vapour-pressure of aqueous solutions by measuring the quantity of water absorbed by a stream of dry air, a method which he had himself employed, and in agreement with his own results, finds that the values so obtained agree with those obtained by the barometric method. Tammann, however, regards the author's determinations of the vapour-pressure of solids containing combined water as inaccurate, but without giving any experimental results in support of his conclusion.

Reserving a more detailed reply to Tammann's criticism, the author makes the following observations:—

(1.) The vapour-density d_1 of the uncombined water is given by the known formula of Stefan, $d_1 = c/l \log p/(p - p_1)$, where c is the coefficient of diffusion, and l the length of the open tube. It follows that approximately $d_1 l/c = 2p_1 M/(2p - p_1)$, and for the combined water $d_2 l/c = 2p_2 M/(2p - p_2)$, or $d_1/d_2 = p_1/p_2$, since $(2p - p_2)/(2p - p_1)$ is nearly equal to unity. Assuming the diffusion to be proportional to the vapour-pressure directly, and to the square of the absolute temperature, this relation is obtained still more directly.

(2.) The author's method of observation determines the constant pressure during dissociation, whilst the statical method gives the final pressure resulting from the dissociation. The method employed by Tammann approximates to the latter when the current of air is slow.

(3.) The author's results agree well together, and warrant the conclusion that the dissociation pressure is constant for a given salt.

(4.) The values obtained for the vapour-pressure vary according to the nature of the salt, by evaporation of part of the water of crystallisation, and by long exposure to the air, which even when there is no sensible evaporation makes the salt much more readily decomposable.

(5.) In the case of chrome alum and copper sulphate, the author has confirmed the values obtained for the vapour-pressure by direct observations on the atmosphere in contact with the salt.

G. W. T.

Some Properties of Solutions. By P. Duhem (*J. de Phys.* [2], **7**, 5—25).—In his treatise "Le Potential Thermodynamique et ses Applications," the author deduced from the researches of Malaguti and of Berthelot, a proposition with regard to the thermodynamic potential Φ, of a solution containing a mass m_0 of solvent and m_1, m_2,

of different substances in solution, a relation which in the case of only two substances in solution is represented by the equation—

$$\frac{d^2\Phi}{dm_1 dm_0} = \frac{d^2\Phi}{dm_2 dm_0},$$

but further investigation has shown that this law is not in agreement with experiment.

The author defines a series of substances 1, 2, &c., of molecular weights w_1, w_2, &c., as belonging to the same series relative to a solvent, provided that when masses m_1, m_2 are dissolved in a mass m_0 of the solution, the equation—

$$w_1 \frac{d^2\Phi}{dm_1 dm_0} = w_2 \frac{d^2\Phi}{dm_2 dm_0},$$

holds good, and he finds from experiment, that all the substances which are soluble in a given solvent can be arranged in a small number of such series. The function $d\Phi/dm_n$ the author calls the thermodynamic potential function of the substance n within the solution. It is then shown to follow very simply that the potential function of the solvent is unaffected by replacing a given weight of one of the substances in solution by an equivalent weight of another substance of the same series. As an important special case of this we have the proposition that if n solutions are formed with the solvent, each containing one of the substances $1, 2, \ldots . n$, belonging to the same series with respect to the solvent, the potential function of the solvent will have the same value in each of the solutions, provided their concentrations are in the proportion of the molecular weights of the substances in solution.

Now it is shown in the author's paper on osmotic pressure (this vol., p. 1022) that on approximation to the point at which the compressibility of water may be neglected, the osmotic pressure expressed in terms of a column of water at the temperature of observation, measures the difference, at this temperature, between the potential function of pure water and that of the water within the solution, each being taken at the pressure in the solution. It follows, therefore, that all the n solutions of the preceding paragraph will be isotonic.

It is then shown that these solutions will have the same solidifying point, a statement which sums up the extensive series of experimental results obtained by Raoult (Abstr., 1880, 523; 1882, 1260; 1883, 7, 278, 952; 1884, 254, 701, 808, 952, 1248.)

The author shows that the remarkable relation between the lowering of the solidifying point of water by dissolving an acid or a base in it, and the energy of the acid or base, first deduced experimentally by Raoult, is to be expected from theoretical considerations.

Kirchhoff ("Ueber einen Satz der mechanischen Wärmetheorie, &c.," *Ann. Phys. Chem.*, **103**), bases his discussion of the properties of gaseous solutions on the three hypotheses: (1) that the gas obeys Boyle's and Gay-Lussac's (Charles's) laws; (2) that the solution of the gas in the liquid does not alter the vapour-pressure of the latter; (3) that the volume of gas dissolved, measured at the final pressure, is at any given temperature in a constant ratio to the volume of the

liquid. The author shows that the second hypothesis is a necessary consequence of the first and third. He then shows that all slightly soluble gases satisfying (1) and (3) belong to the same series, and that for all gases of this kind and other substances belonging to the same series—

$$\frac{\Pi}{s} = \frac{RT}{w_1},$$

where s is the concentration or ratio of mass of dissolved substance to mass of solvent, w_1 the molecular weight of the former, T the temperature, and Π the osmotic pressure of the solution expressed in terms of a column of the solvent.

This law, the form of which recalls the general formula including the laws of Boyle, Gay-Lussac (or Charles), and Avogadro, is the one given by Vant' Hoff as being applicable to the majority of substances in sufficiently dilute solutions.

The author further finds that for solutions of gases obeying the conditions (1) and (3), there is absolutely no disengagement of heat due to dilution or to solution of the gas.

In general, where $\lambda_1 m_1$ is the quantity of heat produced by the solution of the mass m_1 of gas, s the concentration of the saturated solution of gas at temperature T, α the coefficient of solubility, namely, the ratio of the volume of the gas to that of the liquid, w_1 the molecular weight of the gas, J the mechanical equivalent of heat, the author obtains the relation—

$$\lambda_1 = -\frac{JRT^2}{w_1}\frac{d}{dT}\log S,$$

which may be transformed by considering properties of gases, to—

$$\lambda_1 = \frac{JR}{w_1 T}\left(1 - T\frac{d\log\alpha}{dT}\right),$$

which is the same as that arrived at by Kirchhoff in the memoir previously referred to.

Similar reasoning may be applied to the case of solutions of salts belonging to the same series as the gases considered, which Raoult and Vant' Hoff call the normal series, giving the results :—

(1.) The dilution of a solution of a substance belonging to the normal series gives rise to no production or loss of heat.

(2.) The heat of solution of any substance belonging to the normal series is independent of the concentration of the solution, and is given by the formula—

$$\lambda_1 = -\frac{JRT^2}{w_1}\frac{d}{dT}\log S,$$

where S is the solublity of the salt at the temperature T, and w_1 its molecular weight.

This law had been given previously by Le Chatelier (Abstr., 1885, 340, 473), as a general formula for heat of solution. The present investigation shows that it is only applicable to substances belonging to the normal series. G. W. T.

Theory of Liquids. By D. KONOWALOFF (*Zeit. physikal. Chem.*, **2**, 1—5).—In a former communication (*Zeit. physikal. Chem.*, **1**, 39), the author showed that if the expansion by heat of a liquid follow the law $v = 1/(1 - kt)$, the alteration in the compressibility with temperature would be expressed by $\phi = \phi_0 T/T_0(1 - kt)^3$. Calculations with this formula by Grimaldi for ether, pentane, and chloroform at 100°, led to values of ϕ totally different from those observed, but it is here shown that this was owing to an error in the calculation, and that whereas in the case of ether and pentane there is certainly a considerable difference between the calculated and observed values, in that of chloroform the two are in close agreement. The explanation of the exception exhibited by ether and pentane is that at a temperature so far above their boiling points the relation $v = 1/(1 - kt)$ no longer holds. The author shows that it may be replaced by $v = (1 - k_1t)(1 - 2k_2t)$ from which $\phi = \phi_0 T(1 - k_1t)/T_0(1 - 2k_2t)^3$, which formula gives values for ϕ for ether and pentane at 100° in close agreement with those observed. H. C.

Attempt to eliminate the Influence of the Change in the Volume of the Vessel when measuring the Compressibility of Liquids. By J. J. BOGUSKI (*Zeit. physikal. Chem.*, **2**, 120—123).—The principle of the method used is to measure the volume of the liquid in compression and in its normal condition, not as usually occurs in one and the same vessel, but in two different vessels. The apparatus described admits of the above being carried out, and a measurement gave for the true compressibility of ether at 19·2° 0·00018346 per atmosphere. H. C.

Compressibility of Sylvin, Rock-salt, and Aqueous Solutions of Potassium Chloride. By W. C. RÖNTGEN and J. SCHNEIDER (*Ann. Phys. Chem.* [2], **34**, 531—551).—In this paper the authors tabulate the results of a large number of experiments in continuation of their researches on compressibility. In a former paper (this vol., p. 22), the authors obtained the value 0·049 for the relative apparent compressibility of rock-salt at 17·9°. They now find by experiments carried out with greater accuracy, that its value is 0·0477 at 18·4°, and that a more exact determination of the content of the piezometer and of the density of the rock-salt used in the former experiments gives the number 0·0475 as the corrected result, and this is in close agreement with the more recent determination. Taking 0·0476 as the value at 18°, and multiplying by 0·000044 Atm.$^{-1}$, the value obtained for the apparent compressibility of water, they obtain for the apparent compressibility of rock-salt the value 0·00000210 Atm.$^{-1}$.

Experiments on powdered rock-salt and powdered sodium chloride show that the compressibility of the powder is slightly greater than that of the solid salt.

For the apparent compressibility of a concentrated solution of commercial chloride of sodium at 18·4° they obtain the value 0·00000284 Atm.$^{-1}$, and for solid sylvin at 18° they obtain the value 0·00000424 Atm.$^{-1}$. The difference between the values of the com-

pressibility of rock-salt and sylvin is a good deal larger than would result from Voigt's researches (*Berl. Ber.*, 1883, 971); his values, however, were not determined directly, but involved certain assumptions of Poisson's with respect to the theory of elasticity.

In a former paper (*Ann. Phys. Chem.* [2], **29**, 193), the authors found that the observations could be represented by the formula $(y - b)(n + a) = (1 - b)a$, where a and b are constants, y the relative apparent compressibility, and n the number of molecules.

If this formula held good for $n = \infty$, we should have $y = b$ as the relative apparent compressibility of the solid salt. In the case of sodium chloride, the results obtained in this manner agree well with the direct determinations, but this is not the case for potassium chloride.

The authors merely publish the results obtained in this paper as an instalment of an extensive series which they hope will ultimately throw some light on the nature of solutions. G. W. T.

Theory of Solution. By W. Ostwald (*Zeit. physikal. Chem.*, **2**, 36—37).—The analogy which Van't Hoff has drawn between a substance in the gaseous state and the same in solution, and the hypothesis of Arrhenius (this vol., p. 896) that substances in dilute solution undergo dissociation into their ions, may be tested by the application of gaseous laws to the state of solution. Such a law is that for dissociation at constant temperature, where neither of the products of decomposition is present in excess, $p/p_1^2 = c$, where p is the pressure of the undecomposed, p_1 that of the decomposed portion, and c a constant. Since pressure in solution is proportional to the amounts u and u_1 of substances present and inversely to the volume, we have $uv/u_1^2 = c$. But u and u_1 may be calculated from the electrical conductivity as Arrhenius has shown. If u_v is the conductivity for volume v of an electrolyte, and μ_∞ the limiting value of the same for infinite dilution, then $u/u_1 = (\mu_\infty - \mu_v)/\mu_v$, since the conductivity μ_v is proportional to the dissociated amount of the electrolyte u_1. Hence for all binary electrolytes we have as the law of dilution $(\mu_\infty - \mu_v)v/\mu_v^2 = \text{const.}$

The author proposes to subject this theory to further investigation. H. C.

Hydrates of Gases. By Villard (*Compt. rend.*, **106**, 1602—1603).—The gases were introduced, together with a small quantity of water, into the tube of a Cailletet's apparatus, compressed, released suddenly, and again compressed, or simply compressed, the tube being cooled below 0°. The latter method was found to be sufficient; the sudden release of pressure only promotes the formation of the hydrates by producing small quantities of ice. So long as the pressure is maintained the hydrates are stable, provided the temperature does not exceed a certain limit, but if the pressure is removed the hydrates decompose with effervescence. On restoring the pressure, the hydrates are again formed on the sides of the tube owing to the moisture having been deposited thereon. The tension of dissociation of several hydrates was determined.

Methane.—This hydrate is formed at −2° to −3° under a pressure

of 30 atmos. Tension of dissociation 27 atmos. at 0°, 200 atmos. at 19°. Above 21° the hydrate decomposes even under a pressure of 300 atmos.

Ethane.—6 atmos. at 0°, 28 atmos. at 12°, above which it decomposes under any pressure.

Acetylene.—7 atmos. at 0°, 34 atmos. at 14°.

Nitrous oxide.—10 atmos. at 0°, 43 atmos. at 12°, above which it decomposes under any pressure.

Hydrates of gases have a critical temperature of decomposition above which they cannot exist under any pressure. Some gases, such as methane or ethane, form crystalline hydrates at temperatures above their points of liquefaction, and the critical temperature of the hydrate is higher than the critical temperature of the gas.

Nitrogen, oxygen, carbonic oxide, nitric oxide, cyanogen, ammonia, propylene, butylene, and allylene do not yield crystalline hydrates under the conditions described. C. H. B.

Hypothesis of Dissociation of Salts in very Dilute Solutions. By E. WIEDEMANN (*Zeit. physikal. Chem.*, **2**, 241—242).—The author criticises the results obtained by Arrhenius and Planck (this vol., pp. 895, 896). The exceptions to the law of the reduction of the freezing point E in water, which is of the form $E = \text{const. } n_1/n$, where n_1 is the number of molecules of the dissolved substance and n that of the solvent, are explained by Planck as arising from the increase in n_1 due to dissociation of the salt molecules, but would equally result from a decrease of n caused by aggregation of the molecules of the solvent at the freezing point. The simplest explanation of the maximum density and expansion of water between 4° and 0° is that the water already contains in a process of formation solid producing ice molecules, so that at the freezing point the salt is no longer in the presence of simple water molecules but of water and ice molecules, and n thus decreasing in value has the same effect on E as an increase in n_1, which, however, remains the same. The anomalous position occupied by water among liquids in respect of density explains the occurrence of exceptions with this solvent to the law of the reduction of the freezing point and not with others. Hence, while still admitting the possibility of a partial dissociation of some salts of weak acids and bases in solution, the above exceptions should be regarded as due in the majority of instances to the behaviour of the solvent and not to that of the salt. Arrhenius looks on the properties of many compounds in solution being of a so-called additive nature, that is, they may be represented as the sum of the properties of the atoms, as strong presumptive evidence of dissociation, but additive properties occur equally in solids, the molecular heat being the sum of the atomic heats, the molecular volume the sum of the atomic volumes, and so on. Those proofs also which are deduced from the electrical conductivity must be considered as unsatisfactory, so little being known at present of the apparently complicated laws by which this process is regulated.

Ostwald, in a note on the above, raises the objection to Wiedemann's assumption with regard to the reduction of the freezing point, that unless it underwent a polymerisation simultaneous and exactly

proportional to that of the solvent, no salt could behave in a normal manner in water, as it is well known that all non-electrolytes do. He points out that Arrhenius has not advanced the view that a compound having additive properties is a sign of dissociation, but only that if substances are dissociated that will have additive properties similar to those which hold for salts in dilute solution. He further maintains that the explanations afforded by the new hypothesis itself serve in great measure to throw light on the nature of the electrical conductivity of solutions, and remove the complications to which Wiedemann alludes. H. C.

Dissociation of some Alums and of Sodium Acetate. By W. Müller-Erzbach (*Ber.*, **21**, 2222—2224).—Potash alum loses 8 mols. H_2O at 44·8° under a constant tension of 0·14, 1 mol. H_2O is given off at 44·2 under a somewhat less constant tension of 0·02 to 0·03, and 3 mols. remain, so that there are certainly two and probably three different degrees of force with which the water is held in combination. Ammonium alum behaves similarly, 8 mols. H_2O are evolved at 41·3° under a tension of 0·11, and 1 mol. H_2O at 45·8 under a tension of 0·016.

Soda alum and chrome alum lose only half of their water under a considerable tension which, for soda alum is 0·88 at 30·5° and for chrome alum 0·49 at 27·8°. With freshly crystallised soda alum at 18·5°, the tension remained 0·05 for two days, rose on the third day to 0·81 at 28·8°, and after that was normal and remained so even at a lower temperature.

Sodium acetate loses all its water at 29·8 under a constant tension of 0·31. F. S. K.

Osmotic Pressure. By P. Duhem (*J. de Phys.* [2], **6**, 397—414).—In a previous memoir in the same volume, the author arrived at some conclusions apparently at variance with those of Van't Hoff (this vol., p. 778), but he now points out that this was due to his having attributed a different meaning to the term osmotic pressure.

When an aqueous solution of salt is separated from pure water by a diaphragm permeable to water but not to the salt, there will be a difference of pressure on opposite sides of the diaphragm when the condition of equilibrium, in which there is no flow of water through the diaphragm, is attained. This difference of pressure, which is a function of the concentration of the solution, Van't Hoff calls the osmotic pressure.

The author then proceeds to apply the theory of the thermodynamic potential to osmotic relations, and deduces some general results of considerable interest, more especially as they confirm a number of conclusions at which Van't Hoff arrived experimentally. He shows that in order to maintain a given solution in osmotic equilibrium with pure water at the pressure p, a pressure p' must be applied to it, depending on p, on the temperature, on the nature of the salt, and on the concentration of the solution, but independent of the shape of the containing vessel, or of the form and material of the diaphragm.

Adopting Willard Gibbs's definitions of the potential function of

pure water and of the salt in the solution, he finds that the pressure p' is the pressure at which the potential function of the water in the solution becomes equal to the potential function of pure water at the pressure p. Both these propositions were formulated by Van't Hoff, and are now shown to follow rigorously from the laws of thermodynamics.

The pressure p' is shown to be necessarily greater than the pressure p, and it must increase with the concentration.

If the compressibility of water is neglected, it is shown that to this approximation the osmotic pressure expressed in terms of a column of water at the temperature of observation, measures the difference, at this temperature, between the potential function of pure water and that of the water within the solution, each being taken at the pressure in the solution.

When two solutions of different salts and of different concentration are in osmotic equilibrium with water at the same pressure p, while the solutions are subject to the same pressure p', Van't Hoff calls them isotonic. The author obtains an expression by means of which, when the vapour-pressure of a solution is known, and also the law of the compressibility of water-vapour, the vapour-pressure of an isotonic solution can be determined. Assuming the laws of Boyle and Gay-Lussac to be applicable to water-vapour, and neglecting the specific volume of the solution in comparison with the specific volume of its vapour, the author arrives at the result that two isotonic solutions at the same temperature have approximately equal vapour-pressures.

Van't Hoff states the theorem as an exact one, but the conditions of his experiments were such that the pressure p', at which the solutions are isotonic, was equal to the vapour-pressure P of one of them, and the formulæ in the present paper will then give P equal to P_1, the vapour-pressure of the other solution.

The author shows that the difference in the solidifying points of two isotonic solutions under any given pressure is exceedingly small. Van't Hoff enunciates this law in an exact but more restricted form, viz., two isotonic solutions have the same solidifying point at the pressure for which they are isotonic, and the author's formulæ show this to be rigorously true.

For the relation between the osmotic pressure $\varpi = p' - p$, and the temperature T, Van't Hoff gives $d\varpi/d\mathrm{T} - \varpi/\mathrm{T} = 0$, but this the author shows to be incorrect, the true relation being of a much more complicated character, and not approximating to this even when, as Van't Hoff supposes, the solutions are so dilute that the further dilution produces no calorific effects. G. W. T.

Permeability of Vegetable Membranes for Air. By E. Lietzmann (*Bied. Centr.*, 1888, 497).—Cork lamellæ, even when there is a great difference of pressure, are impermeable for gases, whilst the tender cellular membrane of the leaves of *Peperomia magnolifolia*, and also the *cuticula* in a saturated state, are permeable to air. Air permeates more readily through a saturated than through an air-dry membrane. The membranes of woody tissue are also very readily

permeated, and the more so if they be moist. Experimenting with bladder, the author finds that animal membranes follow the same law as vegetable. E. W. P.

Equilibrium in the Retention of Water by Dilute Sulphuric Acid and Hydrated Salts. By W. Müller-Erzbach (*Zeit. physikal. Chem.*, **2**, 113—119).—Two salts were experimented with, sodium dithionate and lead acetate. In each case water passes from the salt to sulphuric acid of a tension that is slightly lower, but the reversal of the change and the passage of water from the acid to the salt requires that the tension of the acid should be considerably higher than that of the salt. This is independent of the state of aggregation of the salt. Experiments were made towards ascertaining the influence which slight impurities might have on the tension, but without definite results. H. C.

Studies in Chemical Dynamics: Oxidation and Reduction Processes. By W. Ostwald (*Zeit. physikal. Chem.*, **2**, 127—147).—The author finds that the increase in the velocity of oxidation and reduction processes produced by the presence of free acids is in proportion to the constants of affinity of these latter. The increase in speed of the reactions $HBrO_3 + 6HI = HBr + 3H_2O + 6I$ and $2CrO_3 + 6HI = 2Cr(OH)_3 + 3I_2$ in the presence of various acids was measured. A comparison with the constants of affinity and the electrical conductivities shows that the numbers representing this increase of speed for each acid are of the same order.

An exception in the first reaction was found for the case of chromic acid. The number for this is abnormally high, the explanation probably lying in a contact action of some sort occurring. That a recurring reduction and oxidation of the chromic acid does not take place is shown by the completely neutral action of chromium sulphate. An influence which may, however, be in some degree susceptible of the latter explanation is that of ferrous and ferric salts, both of which increase the speed of the reaction. In the case of ferrous salts the increase appears to be approximately inversely proportional to their molecular weights. A large number of chlorides were examined but were mostly indifferent, having no appreciable effect on the speed of the reaction. Manganese chloride increases the speed slightly, and cupric chloride very appreciably, but this latter oxidises the hydriodic acid. Cadmium chloride has a great effect in decreasing the speed, but this is owing to its decomposition by the hydriodic acid and consequent removal of the latter. The description of the behaviour of the chlorides applies equally to that of the bromides. H. C.

Decomposition of Ammonium Salts by Bromine Water. By S. Raich (*Zeit. physikal. Chem.*, **2**, 124—126).—The decomposition of ammonia by bromine, $2NH_3 + 3Br_2 = N_2 + 6HBr$, takes place with very different velocities for ammonium salts and for free ammonia, being much slower with the former. As this would probably depend in some measure on the affinity coefficients of the acids, experiments were made to determine the relative velocities

of the reaction with different ammonium salts. The chloride, nitrate, sulphate, oxalate, acetate, tartrate, monochloracetate, formate, succinate, and malonate were taken in 1/20 normal solutions, and 12 c.c. of each titrated with 25 c.c. 3/40 normal bromine water at 25°. The results show that the velocity of the reaction is in some way connected with the affinity coefficients of the acids, as the stronger the acid which is combined with the ammonia the slower the reaction. In the case of the oxalate, formate, succinate, and malonate, the bromine attacks the acid as well as the ammonia, thus complicating the reaction. H. C.

Velocity of Etherification determined by means of Electrical Conductivity. By NEGREANO (*Compt. rend.*, **106**, 1665—1668).—The rate of etherification of a mixture of ethyl alcohol and acetic acid was measured by determining the resistance of the liquid by Lippmann's method. The results were similar to those of previous observers, and the method is generally applicable. C. H. B.

Influence of Neutral Salts in Chemical Reactions. By J. SPOHR (*Zeit. physikal. Chem.*, **2**, 194—217).—The author has continued his investigations on the influence of neutral salts on the inversion of cane-sugar (Abstr., 1885, 1181; 1886, 502) by studying the influence which the chlorides of potassium, sodium, lithium, magnesium, cadmium, mercury, calcium, strontium, barium, and manganese have on the rate of the inversion of cane-sugar by hydrochloric acid. This influence appears as before to be proportional to the amount of neutral salt present, and for salts belonging to the same series in the periodic system to decrease with rising molecular weight. The same amount of neutral salt with strong acid has approximately as much greater influence than with weak acid as the constant of inversion of the former is greater than that of the latter, so that the influence of any given quantity of neutral salt will decrease with the increasing dilution of the acid.

A study was also made of the influence of salts on the saponification of ethyl acetate by potassium hydroxide. From these and the above experiments the author draws the conclusion that the influence of the neutral salt is due to its taking part in the reaction, and explains how this may be assumed to occur on the Williamson-Clausius hypothesis of dissociation. H. C.

Freezing Mixtures containing Solid Carbonic Anhydride. By L. CAILLETET and E. COLARDEAU (*Compt. rend.*, **106**, 1631—1634).—The temperatures were measured by means of a thermoelectric couple which had been graduated against a hydrogen thermometer.

Compressed or porous solid carbonic anhydride alone, under atmospheric pressure, gives a temperature of about −60°; in a vacuum maintained by means of a pump and potash the temperature is −76°. A mixture of ether and solid carbonic anhydride has a temperature of −77° under ordinary pressure, and −103° in a vacuum. This mixture solidifies liquid carbonic anhydride.

When solid carbonic anhydride is added to ether, it at first disappears rapidly, not owing to volatilisation, but because it dissolves in the

ether. The ether remains transparent, but after some time bubbles of gaseous carbonic anhydride are given off. If further quantities of the anhydride are added, the liquid becomes saturated, and loses its transparency. The temperature gradually falls until it attains a minimum exactly at the point of saturation. Any further addition of the anhydride causes no further reduction of temperature, but the liquid becomes more and more turbid. It is evident that the effect of the ether is due to its solvent action on the carbonic anhydride. Other solvents producing low temperature with the anhydride are methyl chloride, −82°; sulphurous anhydride, −82°; amyl acetate, −78°; phosphorus trichloride, −76°; alcohol, −72°; and ethylene chloride, −60°. The temperature of mixtures of carbonic anhydride with methyl chloride or sulphurous anhydride in a vacuum is so low that the solvent solidifies, and the temperature of the mass remains constant from this point. With methyl chloride the temperature obtained is −106°. A mixture of carbonic anhydride and chloroform becomes solid under ordinary pressure, and has a temperature of −77°. C. H. B.

Configuration of the Molecule of Benzene. By F. Herrmann (*Ber.*, **21**, 1949—1959).—The author states that the various possible positions in space of the atoms in the molecule of a hydrocarbon can be ascertained by simple geometric considerations without making any assumption as to the nature of the carbon-atom. This statement is discussed and explained with the aid of figures in the case of methane, hexamethylene, and benzene. F. S. K.

The Air of Coal Mines. By T. G. Nasmyth (*Brit. Med. Journ.*, **2**, 1888, 222—229).—The following is a summary of the results obtained from the physical, chemical, and biological observations made from a sanitary standpoint, on the air in coal mines.

The *temperature* was determined by wet and dry bulb thermometers, one set being placed above ground, the other below. Both were read daily for a period of three months, and the results are given in tables. The most noteworthy points were the uniform temperature of the mine from day to day, and the fact that the air was nearly always saturated. On 21 consecutive days, the temperature remained at 53° F.; the highest temperature in the mine was 55° F., the lowest, 53° F.

The *volume* of the air was measured by the anemometer, and tables are given showing the large volumes of air which are passed into mines.

Carbonic anhydride was estimated by Pettenkofer's method. In mines under 100 fathoms deep, the percentage in volumes was 0·181; in deep mines over 100 fathoms, it was 0·219.

Organic matter, estimated by Carnelley's method, showed a high result, expressed as oxygen needed for a million volumes of air, for shallow mines 30, and for deep 39. This high result is probably due to those various chemical substances which decolorise potassium permanganate, beside organic matter.

Oxygen was estimated by Franke's method. This method is not

strictly accurate, and the results are comparative. The estimation showed that when ventilation was deficient, oxygen was diminished.

Estimations were also made of ammonia and albuminoïd ammonia by Wanklyn's method.

In the biological experiments, Hesse's method for collecting and estimating the micro-organisms in air was employed. Some hundreds of microscopic preparations and cultivations were made, and for particulars the original report must be consulted. All varieties of microbes were found, the numbers not always being in relationship to the impurity of the air. Moulds preponderated. The presence of horses and men had always a distinct effect in increasing the numbers in Hesse's tubes. This method, though the best we have, is not exact, as it is impossible to be sure that all microbes are deposited on the nutrient jelly; and also a litre of air run out from the aspirator does not imply that a litre of air has passed into Hesse's tubes.

The general conclusion is drawn that the air of coal mines compares favourably with that of one-room houses, schools, and workshops. Miners are not unhealthy, and are not particularly affected by phthisis or bronchitis. These statements are supported by statistics.

W. D. H.

Inorganic Chemistry.

Molecular Weight of Sulphur, Phosphorus, Bromine, and Iodine in Solution. By E. Paternò and R. Nasini (*Ber.*, **21**, 2153—2155).—The authors have made determinations of the molecular weight of sulphur, phosphorus, bromine, and iodine, by Raoult's method in a similar manner to that described (*Ber.*, **19**, 2527). Benzene was the solvent employed in the case of sulphur, and solutions of various concentration were experimented on. The coefficient of depression was constant, and the molecular formula was found to be S_6. Aqueous and acetic acid solutions were employed for the determinations with bromine, and the results show that the molecular formula is Br_2. The molecular formula of iodine was found to be I_2, when very dilute solutions of benzene are employed, but in more concentrated solutions the results seem to indicate a greater complexity of the molecule. The molecular depression is constant in acetic acid solutions, and the results obtained point to molecular formula intermediate between I and I_2. From the results obtained with phosphorus in benzene solution, it seems probable that it contains a mixture of molecules of P_4 and P_2, but the sample employed was not quite pure.

F. S. K.

Molecular Weight of Sulphur. By H. Biltz (*Ber.*, **21**, 2013—2017).—The author made determinations of the vapour-density of sulphur by Meyer's method, but obtained no constant results, although the apparatus was filled with pure nitrogen, and experiments with mercury gave fully concordant determinations. The cause of this

disagreement is the presence of an indifferent gas, and it was found that the larger the quantity of sulphur taken the higher were the results obtained.

Determinations made with Dumas' apparatus at 518° gave constant results, but the density gradually decreases as the temperature increases. At 467·9° the vapour-density is greater than that calculated for S_6, and there is no fact which points to the existence of such a molecule. There is just as much ground for assuming the existence of molecules of S_5, S_7, S_2, or S_n, as of S_6.

The only results which are constant within a very considerable range of temperature are those which agree with the molecular formula S_2. F. S. K.

Remarks on the preceding Article. By V. Meyer (*Ber.*, **21**, 2018—2019).—The results obtained in the previous paper show clearly that in vapour-density determinations made by the author's method, the quantity of substance taken and the size of the bulb are without influence on the result only when the substance, at the temperature of the determination, is a normal gas, the density of which is not changed by pressure or temperature. In other cases, the results are dependent on the relative quantities of the substance and the gas (air or nitrogen) with which its vapour is mixed. F. S. K.

Property of Carbon similar to that of Spongy Platinum. By G. A. Hirn (*Compt. rend.*, **106**, 1784—1785).—The author has noticed that a heated thread of carbonised lamp wick occasionally remained incandescent in a mixture of alcohol vapour with air. C. H. B.

Absorption of Carbonic Oxide by Cuprous Chloride. By H. Drehschmidt (*Ber.*, **21**, 2158—2164; compare this vol., p. 88).—A reply to Hempel (this vol., p. 556). A hydrochloric acid solution of cuprous chloride, free from carbonic oxide, and almost saturated with nitrogen by keeping it in an atmosphere of the latter, does not evolve gas when placed in contact with hydrogen, but the volume of hydrogen (89·71 parts) diminishes slightly (0·55 part). In a second and third experiment with the same volume of hydrogen, the diminution was 0·23 and 0·17 part respectively, and if the solution was now brought into contact with the same volume of nitrogen, the latter increased by 0·17 part.

When 92·95 parts of coal-gas, freed from carbonic anhydride, heavy hydrocarbons, and oxygen, is treated with 10 c.c. of a freshly prepared hydrochloric acid solution of cuprous chloride, and the process repeated five times, the loss of volume is 8·35, 1·63, 0·41, 0·27, and 0·03 parts respectively, the consecutive diminutions being due to absorption of carbonic oxide. Experiments in which carbonic oxide was absorbed by a solution of cuprous chloride, the solution then shaken with pure nitrogen, and the resulting gaseous mixture analysed, show clearly that the solution evolves carbonic oxide when placed in contact with nitrogen. F. S. K.

Reactions Occurring in the Preparation of Water and Heating Gas. By J. Lang (*Zeit. physikal. Chem.*, **2**, 161—183).—The object in these investigations was to ascertain the nature of the reactions which occur at different temperatures between the gases produced by the action of steam on carbon and the limits of such reactions. The action of methane on carbonic anhydride sets in at about 700°, a partial reduction taking place, carbon being deposited, and carbon monoxide formed. Some of the carbon is due to the decomposition of the methane by heat which occurs at this temperature. Hydrogen is the chief product of the action of water-vapour on methane at about 1000°, arising both from the action of heat on the methane, and from the reaction between the two gases. The carbon from the methane gives a hard, bright deposit of graphite, which reduces the carbonic anhydride formed in this reaction but slowly even at 1000°, the carbon monoxide present being mostly due to the reduction of the dioxide by the methane. The action of oxygen on carbon results in the formation of carbonic anhydride alone if conducted at 500°, and not too rapidly. A rapid action causes local heating of the carbon and consequent reduction of a small portion of the dioxide to monoxide, which is also brought about by raising the temperature of the reaction. If oxygen mixed with carbon monoxide be passed over carbon, no change takes place in the monoxide at 500°, if the reaction takes place slowly, as the temperature is not high enough for its combustion, but the carbon is burnt to dioxide by the oxygen. If the stream of mixed gases be passed rapidly, the heat of the latter reaction raises the temperature, and combustion of the monoxide then takes place. These experiments show that in the combustion of carbon the formation of the monoxide is preceded by that of the dioxide. The action of water-vapour on carbon begins at about 600°. At this temperature carbonic anhydride and hydrogen are the sole products, but as the temperature rises carbon monoxide is also formed from the reduction of the dioxide. This is somewhat decreased in amount by the opposing action of the water-vapour, $CO + H_2O = CO_2 + H_2$, an effect which may be much reduced, though not entirely overcome, by having the carbon porous and present in quantity, offering a large surface for the quick removal of the water-vapour. The reduction of carbon dioxide to monoxide, the action of water-vapour on carbon monoxide, and the decomposition of water-vapour by carbon, can only take place within certain limits strictly defined by temperature, and at no temperature is any one of these reactions a complete one. H. C.

Lowest Oxide of Silver. By O. v. d. Pfordten (*Ber.*, **21**, 2288—2299).—The compound described as oxide (compare Abstr., 1887, 699, and this vol., p. 221) must be regarded as a hydrate of silver, the composition of which is probably Ag_4H_2O (? $Ag_4{}'H{\cdot}OH$). The product which is first precipitated in the tartaric acid reaction (*loc. cit.*) is the hydrate, containing silver tartrate or sodium tartrate; it retains its impurities when washed with alcohol, alcoholic acetic acid, or solutions of different salts, and contains about 4 per cent. of water. The flocculent precipitate produced on adding alkali is

probably the pure hydrate, but it cannot be obtained in the dry state free from impurities; when washed with aqueous or alcoholic potash or ammonia, or with acetic acid, the organic impurities are removed, and a black powder is obtained which contains varying quantities of water, but always less than 4 per cent. Both preparations contain extremely small quantities of oxygen.

Silver hydrate is converted into metal when treated with hydrochloric acid, sulphuric acid, and most other acids, but phosphorous and sulphurous acid have no action in the cold. It is much more readily oxidised by potassium permanganate, potassium ferrocyanide, potassium dichromate, and iodine than metallic silver, and is decomposed into metal and water by cold, concentrated hydrogen peroxide, or when washed with water or solutions of salts. It loses its water slowly at 100°, but quickly at 110°.

All black compounds which are obtained by reducing solutions of silver probably consist of the hydrate. F. S. K.

Solubility of Calcium Compounds. By A. H. Allen (*Chem. News*, **57**, 236).—The author points out that the solubility of many calcium salts diminishes with an increase of temperature; such is the case, for example, with the acetate, the propionate, the normal valerate and butyrate, the iso-octate, the citrate, and the benzoate. This property, therefore, appears to be connected with the basic radicle as well as with the decomposition of unstable hydrates.

D. A. L.

Constitution of Hydraulic Cements. By H. Le Chatelier (*Annales des Mines*, 1887, 345—404).—The author shows that when gypsum is subjected to a very regular and progressive rise of temperature, dehydration takes place in two stages; the first, at 120—130°, results in the loss of 1·5 mol. H_2O; the second, at 160—170°, completes the dehydration. In confirmatory experiments, gypsum was exposed for prolonged periods to various fixed temperatures, and it was observed that even at 155° the dehydration did not extend beyond the first stage, whilst at 194° dehydration was complete. The decomposition is independent of pressure, but proceeds more or less rapidly according as the temperature is higher or lower. The product of the first stage, $2CaSO_4,H_2O$, may be obtained in long, rectangular prisms of extreme tenuity, by heating a saturated solution of calcium sulphate at 130—150° in sealed tubes, then to avoid rehydration, which takes place very rapidly in water below 130°, the tube is opened while hot, and the moist contents are thrown into absolute alcohol; the crystals obtained in this manner when dried at 100° have the above composition. The incrustation in boilers supplied with sea-water is an impure form of this compound. The author explains the setting of plaster, and bases his explanation on the hydration of the burnt gypsum in the presence of water, the formation of a supersaturated solution of normal sulphate, and the crystallisation of the latter salt; the water set free is then ready to attack a fresh portion of burnt gypsum, and so on until the whole mass has undergone conversion. Under these conditions, the sulphate crystallises abnormally, forming spherulitic

aggregations of filiform prisms; favourable both for great adhesion and interlacing, as well as for the formation of a solid crystalline mass.

In support of this explanation the following experiment was made with anhydrous sodium sulphate, which when powdered and mixed with water sets like plaster, but being more soluble is more suitable for the experiment. A glass tube, divided midway by means of a piece of metallic gauze, was filled with water, and pieces of fused sodium sulphate were placed on the gauze; the tube was then plunged into cold water to prevent rise of temperature. After a few hours a solid mass of hydrated sodium sulphate had formed at the bottom of the tube several centimetres away from the anhydrous salt, showing that the setting can take place away from the mass, and is due to the formation of, and crystallisation from, a supersaturated solution.

Investigations on barium cements led to the production of the anhydrous silicates $BaSiO_3$ and Ba_2SiO_4 by heating together the requisite proportions of silica and barium oxide. The fused mass obtained in the first case, when examined in section under the microscope, appears as an entanglement of long prismatic crystals, with slight double refraction and easy cleavage. The second silicate simply cakes together, and forms a porous mass exhibiting only a confused crystallisation under the microscope. Both these set when mixed with water, owing to the formation of the hydrated silicate $BaSiO_3,6H_2O$, which is likewise formed when pulverised calcined silica and barium hydroxide are quenched with water, and when a soluble silicate is added to baryta-water; in the latter case, it forms a bulky precipitate, which gradually becomes crystalline, and after 24 hours is entirely changed into tabular crystals, identical with those formed in glass bottles containing baryta-water.

The author has made extensive and elaborate investigations on calcareous cements, in the course of which he has made a careful study of the decomposition of calcium carbonate by heat; all details of the methods and apparatus employed are given in the original. When heated rapidly, decomposition starts from 890° to 930°, according as the carbonate is precipitated carbonate, chalk, or marble; but if it contains magnesia the decomposition commences at 720°. In more careful experiments it was observed that at the same temperature the tension limit is the same for precipitated carbonate, chalk, or marble, but that the limit is reached with greater rapidity by the precipitate. Below 500°, the decomposition is too slow to admit of accurate experiments; at 500°, the limit is not reached until after two hours, and then only equals $\frac{1}{1000}$ of the mass experimented on per hour, and a temperature of 812° is required before the tension equals atmospheric pressure.

According to the author's experience, calcium hydroxide, when heated rapidly, decomposes at a temperature of 530° to 540°; when carefully heated below 100°, there is no appreciable decomposition; at 350°, the tension of dissociation is only 100 mm. of mercury, and only reaches one atmosphere at 450°. It may, therefore, be considered as quite stable in dry air at ordinary temperatures, and moreover it is only attacked very slowly by carbonic anhydride.

The author has prepared and fully described the anhydrous silicates $CaSiO_3$, Ca_2SiO_4, $Ca_3Si_2O_7$, Ca_3SiO_5. The first three he prepared by heating together silica and lime in proper proportions; the first is identical with wollastonite, the third is simply a mixture of the first and second. The second forms a hard, solid, fused mass while hot, but as it cools it cracks, swells, and ultimately falls to a white powder, resembling slaked lime; this really consists of microscopic, prismatic, crystalline fragments, slightly doubly refractive, and, at times, striated in the direction of the greatest length. This phenomenon is the result of the dimorphism of this silicate, the change is accompanied by development of heat. The spontaneous pulverisation in some slags and cements is now attributed to the presence of this silicate. This power of pulverisation is diminished by the presence of magnesia, iron oxide, or alumina, hence the pulverisation in slags may be much delayed. With equal molecular proportions of magnesia and lime, the resulting mass remains absolutely compact, hard, and definitely crystallised, it is, in fact, monticellite, $CaMgSiO_4$. The compound Ca_3SiO_5 cannot be obtained in the same way as the others by heating lime and silica together, nor by using a flux such as calcium chloride as well, in fact, in the latter case a calcium chlorosilicate is produced. This substance, $Ca_2SiO_4,CaCl_2$, forms orthorhombic crystals with double cleavage and energetic double refraction; measurements of the crystals are given. The crystals are attacked by water, but not by alcohol. Heated in steam above 450°, it gave rise to an amorphous compound, presumedly Ca_3SiO_5; neither of the preceding silicates set when mixed with water, but this does, forming a hard, durable briquette, which shows conclusively that it not only differs from them, but that it is also free from uncombined lime. The author could not obtain hydrated silicates by direct hydration of the above anhydrous silicates; the only one prepared by him was $CaSiO_3$,Aq (possibly $2\frac{1}{2}$ mols. H_2O) by precipitating lime-water with a solution of silica.

The author has also investigated those calcium aluminates which may be expected to occur in cements, and pursuing the synthetical methods by fusion, as adopted in the case of silicates, he has prepared three anhydrous aluminates, and one hydrated aluminate. $CaAl_2O_4$, corresponding with spinel, crystallises in scarcely fusible, very hard cubic crystals. $Ca_3Al_4O_9$ (the author is not quite certain about the composition of this salt) forms orthorhombic crystals with very strong double refraction, which fuse with moderate facility. $Ca_3Al_2O_6$ forms easily fusible, cubic crystals. When pulverised and quenched with water, all three set like plaster of Paris, but when agitated with excess of water they decompose, both lime and alumina pass into solution, from which the alumina is partly, or if lime water is added, wholly precipitated in the form of a crystalline hydrated calcium aluminate. This salt, $Ca_4Al_2O_7,12H_2O$, is the only hydrated calcium aluminate stable in the presence of free lime, and, therefore, capable of existing in wetted cements; it crystallises in spherulitic groups of long needles, forming compact spherulites when the crystallisation is slow. Calcium aluminates become rapidly less susceptible, and ultimately indifferent to the action of water when magnesium is substituted for calcium. The

author does not describe any definite anhydrous calcium ferrate, and only one hydrated ferrate, a white substance decomposed by water and by carbonic anhydride, but not by lime-water. Carbonic anhydride liberates ferric hydroxide.

Mixing alumina and ferric oxide and then heating with lime increases the fusibility of the corresponding lime salts. The author has obtained the alumino-ferrate, $Al_2O_3,Fe_2O_3,3CaO$, crystallising in well-defined, long, red needles. By fusing calcium ferrates with calcium chloride, brown plates terminating in symmetrical obtuse points are obtained of the composition $Fe_2O_3,CaO,CaCl_2$. They have a remarkably easy cleavage, but are not attacked by water or acetic acid. The aluminates under similar circumstances yield very fusible glassy substances, which are decomposed by water.

The rest of this monograph treats of the character and behaviour of the various constituents of cements when they are present in the burnt cementitious mixtures or in the wetted cements. D. A. L.

Action of Alkaline Phosphates on the Alkaline Earths. By L. Ouvrard (*Compt. rend.*, **106**, 1599—1601).—The oxide or a salt of the alkaline earth was dissolved in the fused phosphate, with or without an alkaline chloride, allowed to cool slowly, and the product treated with water.

Barium oxide dissolves readily in potassium metaphosphate or pyrophosphate, and in both cases yields the pyrophosphate $Ba_2P_2O_7$ in monoclinic prisms which dissolve ready in dilute acids and in concentrated sulphuric acid. In presence of potassium chloride, the result is the same if the proportion of the phosphate is not below 5 per cent., but with a lower proportion a chlorophosphate is formed. Precipitated barium phosphate behaves in the same manner as the oxide. Barium sulphate also dissolves and yields the pyrophosphate, the excess of sulphate crystallising in the form of barytes.

Potassium orthophosphate with barium oxide, chloride, or phosphate, yields the compound $P_2O_5,2BaO,K_2O$ in transparent, dendritic crystals, soluble in dilute acids.

Sodium metaphosphate and pyrophosphate yield either barium pyrophosphate or, if the proportion of barium is considerable, the compound $P_2O_5,3BaO$, which crystallises in large, transparent lamellæ, seemingly belonging to the regular system; sp. gr. 4·1 at 16°. The crystals dissolve in dilute acids and in concentrated sulphuric acid. Sodium chloride promotes crystallisation, but if the proportion exceeds a certain limit, a chlorophosphate is formed. Sodium orthophosphate yields only the compound $P_2O_5,3BaO$. Barium sulphate is not decomposed by the sodium phosphates; it dissolves to a certain extent but crystallises unaltered on cooling.

Calcium oxide, phosphate, or sulphate with potassium metaphosphate or pyrophosphate, yield the compound P_2O_5,CaO,K_2O in large, transparent hexagonal lamellæ derived from the regular octahedron; sp. gr. 2·7. The same compound is also obtained from calcium chloride or fluoride if the alkaline phosphate is in sufficient excess; it dissolves readily in dilute acids. Tripotassium phosphate and calcium oxide form

the compound $P_2O_5,2CaO,K_2O$, which has the same crystalline form as the corresponding barium salt, and has already been described by Grandeau, who obtained it by Debray's method. Sodium metaposphate yields two products according to the proportion of oxide employed, namely, $9P_2O_5,10CaO,8Na_2O$, which was previously obtained by Wallroth under the same conditions, and forms monoclinic lamellæ, and $P_2O_5,2CaO,Na_2O$, which has been described by Ditte, and forms transparent hexagonal rosettes. Sodium pyrophosphate and orthophosphate yield the salt $P_2O_5,2CaO,Na_2O$, and also the salt $2P_2O_5,3CaO,3Na_2O$, which crystallises in slender, transparent, monoclinic needles melting to a white enamel at a red heat. With the carbonate, phosphate, sulphate, chloride, and fluoride, the same products are obtained as with the oxide. With sodium or calcium chloride in excess, chlorophosphates corresponding to apatite and wagnerite are formed.

Strontium oxide and salts with potassium meta- or pyro-phosphate yield the compound P_2O_5,SrO,K_2O, identical in form with the corresponding calcium salt. Tripotassium phosphate produces the compound $P_2O_5,2SrO,K_2O$, identical in form with the analogous barium and calcium compounds. Sodium meta- and pyro-phosphates yield first the compound $P_2O_5,2SrO$ in small, rhombic prisms similar to those of the barium salt, and then the compound $P_2O_5,2SrO,Na_2O$ analogous to the product obtained with calcium. Sodium orthophosphate yields only the salt $P_2O_5,2SrO,Na_2O$; strontium sulphate is not decomposed by the sodium phosphates.

Barium is not readily displaced by alkalis and therefore does not readily form double salts. Calcium forms double phosphates only, and strontium occupies an intermediate position. C. H. B.

Decomposition of Barium Ferrate at High Temperatures. By G. Rousseau and J. Bernheim (*Compt. rend.*, **106**, 1726—1728.)—If barium ferrate is introduced into barium chloride or barium bromide fused at 800—1300°, it rapidly decomposes with effervescence owing to the liberation of oxygen, and leaves a residue of ferric oxide. If, however, about 3 grams of barium ferrate is added gradually to a fused mixture of 15 grams of barium chloride or bromide, and 5 grams of barium oxide, and the product is treated with water, it yields brownish-black crystals of barium ferrite. At 1100°, about 6 grams of barium oxide is required to prevent the decomposition of the ferrite, and at an orange-red heat 12 grams is necessary. It follows from these results that whereas barium ferrate if heated alone decomposes into oxygen, ferric oxide, and barium oxide, when heated with barium oxide it yields oxygen and barium ferrite. The decomposition of the ferrate in presence of neutral or slightly basic fluxes is analogous to the decomposition of alcoholates by dilution, but the latter is progressive whilst the former takes place suddenly as soon as the proportion of barium oxide has fallen below a certain limit. If barium carbonate is present, it crystallises from the oxychloride which is formed, and the crystals are not readily separated from those of the ferrite. C. H. B.

Double Phosphates in the Magnesium Group. By L. OUVRARD (*Compt. rend.*, **106**, 1729—1732.)—These phosphates were obtained in the same way as the phosphates of the barium group (this vol., p. 1033).

Magnesium.—With potassium metaphosphate, the sole product is the compound $3P_2O_5,2MgO,K_2O$, which crystallises in large, monoclinic prisms, very soluble in dilute acids; sp. gr. at 20° = 2·4. It is analogous to the double magnesium sodium phosphate obtained by Fleitmann and Hennerberg in the wet way.

Potassium pyrophosphate or orthophosphate yields rhombic prisms of the salt $P_2O_5,2MgO,K_2O$, previously described by Grandeau. Magnesium phosphate yields the same products as the oxide, but the chloride yields a chlorophosphate unless the alkaline phosphate is present in considerable excess.

With sodium metaphosphate at a low temperature, the magnesium oxide not being in excess, the salt $9P_2O_5,10MeO,8Na_2O$ is obtained in highly macled, monoclinic prisms; sp. gr. at 20° = 2·7. This compound has previously been described by Wallroth. At a high temperature, the metaphosphate yields dendritic crystals of the compound $P_2O_5,MgO,2Na_2O$, and the same product is obtained with the pyrophosphate. It is readily soluble in dilute acids. Sodium orthophosphate yields the salt $2P_2O_5,3MgO,3Na_2O$ in dendritic crystals which depolarise light.

Zinc and cadmium yield compounds which are strictly analogous. With potassium metaphosphate, the salt P_2O_5,MO,K_2O is obtained in highly macled crystals which depolarise light and are soluble in dilute acids. Potassium pyro- or ortho-phosphate yields monoclinic prisms of the salt $P_2O_5,2MO,K_2O$, very soluble in dilute acids. Zinc and cadmium phosphates yield the same products as the oxides, and alkaline chlorides have no effect on the result even when present in large excess. The compound just described can in fact be obtained by the action of cadmium or zinc phosphate on potassium chloride.

Sodium metaphosphate yields the salt P_2O_5,MO,Na_2O, described by Wallroth, or the salt $P_2O_5,2MO,Na_2O$, described by Scheffer, or a third salt, $P_2O_5,MO,2Na_2O$; according to the relative proportions of the oxides; sodium pyro- or ortho-phosphate yields the compounds $P_2O_5,MO,2Na_2O$ and $P_2O_5,2MO,Na_2O$.

Manganese yields products similar to those obtained with zinc and cadmium, but in presence of an alkaline chloride chlorophosphates are formed if the proportion of alkaline phosphate falls below a certain limit. P_2O_5,MnO,K_2O and $P_2O_5,2MnO,K_2O$ crystallise in monoclinic prisms; P_2O_5,MnO,Na_2O forms highly macled rose-coloured prisms which are probably triclinic. $P_2O_5,MnO,2Na_2O$ and $P_2O_5,2MnO,Na_2O$ are isomorphous with the corresponding zinc and cadmium salts.

Cobalt and nickel form strictly analogous compounds. Potassium metaphosphate yields monoclinic prisms of the composition

$$2P_2O_5,3MO,3K_2O,$$

and the pyro- and ortho-phosphate yield rhombic crystals of the salt $P_2O_5,2MO,K_2O$, all soluble in dilute acids. The presence of potassium

chloride promotes crystallisation, but exerts no other influence on the result.

Sodium metaphosphate yields the salt $9P_2O_5,10MO,8Na_2O$ in macled, dichroic violet or rose-coloured prisms, or, if the oxide is in excess, the salt $P_2O_5,2MO_3,Na_2O$, which is isomorphous with the corresponding zinc salt; sodium pyro- or ortho-phosphate yields products strictly analogous to those obtained with zinc, cadmium, and manganese. In presence of sodium chloride, all the double phosphates of nickel or cobalt are converted into the salt $P_2O_5,2MO,Na_2O$.

C. H. B.

Action of Calcium Carbonate on Cadmium Chloride and Cadmium Bromide. By A. DE SCHULTEN (*Compt. rend.*, **106**, 1674 1677).—When cadmium chloride solution is heated with powdered marble in sealed tubes at 200°, some carbonic anhydride is liberated, but no cadmium carbonate is formed. The liquid, however, deposits small hexagonal crystals which dissolve in hydrochloric acid without effervescence. They have the composition $CdCl_2,Cd(OH)_2$, and lose only a small quantity of water at 280°, but become anhydrous at dull redness, the cadmium chloride volatilising to a certain extent. The crystals are hexagonal prisms with transverse striations, terminated by hexagonal pyramids; sp. gr. at 15° = 4·56. The behaviour of the compound when heated indicates that it has the constitution Cd(OH)Cl. Similar crystals are obtained by heating cadmium chloride and cadmium hydroxide in sealed tubes; the iodide and bromide likewise attack the hydroxide. Haberman obtained an amorphous precipitate of the same composition by adding dilute ammonia to a boiling concentrated solution of cadmium chloride.

Cadmium bromide yields a similar compound, Cd(OH)Br, which crystallises in thin, colourless transparent lamellæ; sp. gr. at 15° = 4·87.

Cadmium iodide and calcium carbonate yield no similar product at 200°.

C. H. B.

Hydrochloride of Cupric Chloride. By P. SABATIER (*Compt. rend.*, **106**, 1724—1726).—When hydrogen chloride is passed into a saturated solution of cupric chloride, heat is developed and the solution becomes brown. If the liquid is cooled, it deposits crystals of the hydrate $CuCl_2,2H_2O$, and the mother-liquor is greenish-yellow. On exposure to dry air, the crystals become greenish-blue without losing their transparency. The crystals of the ordinary dihydrate remain unaltered in the mother-liquor of the crystals just described, and the latter owe their different appearance to the presence of some of the mother-liquor.

If the quantity of hydrochloric acid in the solution is increased, the solubility of the cupric salt at first decreases, but afterwards increases, as Engel observed in the case of stannous chloride, and the change in solubility corresponds exactly with the point at which the greenish colour of the liquid becomes reddish. If the reddish-brown solution is cooled to 0°, it deposits radiating, acicular, red crystals of the composition $CuCl_2,2HCl + 5H_2O$, which rapidly change to crystals of the ordinary hydrate even in concentrated hydrochloric acid, or in a

current of hydrogen chloride. They may, however, be preserved in the mother-liquor at the ordinary temperature. They rapidly lose hydrochloric acid when exposed to air and immediately if placed in water or if heated. C. H. B.

Hydrochloride of Cupric Chloride. By P. Sabatier (*Compt. rend.*, **107**, 40—41).—The hydrochloride of cupric chloride described by Engel (this vol., p. 558) is probably identical with that described by the author (preceding Abstract), the difference in the analytical results being due to decomposition during the process adopted by Engel for drying the salt.

The author has determined the variations in the solubility of cupric chloride in presence of hydrochloric acid at 21·5°, the result being stated in equivalents of the salt and acid respectively dissolved in 100 equivalents of water.

Hydrochloric acid	0	0·95	1·76	4·19
Cupric chloride	11·91	10·56	8·36	5·69
Sp. gr.....	1·50	1·47	1·44	1·31

Hydrochloric acid..	6·22	7·45	14·02	15·25	16·95
Cupric chloride....	4·32	3·86	6·46	7·26	8·19
Sp. gr.	1·28	1·27	1·34	1·38	1·43

The variations are much greater at 21·5° than at 0°.

C. H. B.

Mercurous Oxide. By W. Bruns and O. v. d. Pfordten (*Ber.*, **21**, 2010—2013).—Mercurous oxide cannot be obtained free from mercury and mercuric oxide. When mercurous acetate is rubbed in a mortar with an excess of alcoholic potash, the product washed with alcohol, then with ether and dried, the mercurous oxide obtained contains metallic mercury even when the whole process is carried out in absence of light, and mercuric oxide is also present in small quantities. Mercurous oxide similarly prepared from aqueous instead of alcoholic solutions, contains the same impurities but in larger quantities.

Mercurous oxide oxidises to mercuric oxide even at the ordinary temperature, and oxidation seems to take place more quickly in presence of moisture. Dry mercurous oxide is completely converted into mercuric oxide and metal when heated at 100° for some time.

F. S. K.

Phosphates of the Cerite Metals. By L. Ouvrard (*Compt. rend.*, **107**, 37—40).—When the oxides of cerium, lanthanum, and didymium are fused with alkaline phosphates in the manner previously described (this vol., p. 1033), they yield strictly analogous isomorphous compounds. The cerium and lanthanum oxides were quite pure, and the didymium contained only a very small proportion of other oxides. The formulæ and crystalline forms of the products were as follows:—With potassium metaphosphate the tribasic phosphate, M_2O_3,P_2O_5, in monoclinic prisms; with potassium orthophosphate or pyrophosphate, right rhombic prisms of the salt $3P_2O_5,2M_2O_3,3K_2O$. The latter is

converted into the tribasic phosphate in presence of potassium chloride; sp. gr. of the cerium salt 3·8; of the lanthanum salt 3·5. Sodium metaphosphate yields monoclinic prisms of the pyrophosphate, $2P_2O_5,M_2O_3,Na_2O$, described by Wallroth, and then, if a larger proportion of the oxide is used, the tribasic phosphates as in the case of the potassium salts. Sodium orthophosphate or pyrophosphate yields the salt $P_2O_5,M_2O_3,3Na_2O$, which is converted into the tribasic phosphate in presence of sodium chloride.

The simple tribasic phosphates are insoluble in acids, but the double orthophosphates and pyrophosphates are readily soluble.

C. H. B.

Absorption Spectra of Rare Earths. By P. Kiesewetter and G. Krüss (*Ber.*, **21**, 2310—2320; compare G. Krüss and L. F. Nilson Abstr., 1887, 890).—Finely divided yttrotitanite or keilhauite from Arendal was melted in a graphite crucible with flowers of sulphur and soda, the melt well washed with water, and the filtrate treated according to Winkler's method (Abstr., 1886, 985), but no germanium was found. The residue was melted with hydrogen potassium sulphate, extracted with water, the solution acidified with hydrochloric acid, and the bases precipitated as oxalates. The white-grey powder, which remained when the mixture of oxalates was ignited, was dissolved in concentrated nitric acid, the solution freed from nitric acid by repeatedly evaporating, and examined spectroscopically as descibed (*loc. cit.*) with the following results:—

Observed position of maximum darkness.		Observed wave-length. Krüss and Nilson.	For	Intensity of absorption-bands.
Position of of telescope tympanum.	Observed wave-length.			
421	683·1	684·0	Tmα	Very feeble.
519	654·4	654·7	Erα	Feeble.
863	580·3	579·2	Diγ	Moderately feeble.
895	575·3	575·4	D γ	Feeble, more so than $\lambda = 579{\cdot}2$.
1106	542·7	542·6	Xβ	Very feeble.
1128	539·3	539·9	?	Very feeble.
1240	523·0	523·1	Erβ	Tolerably strong.
1254	521·3	521·5	Diδ	Strong, stronger than $\lambda = 579{\cdot}2$.
1589	485·9	485·5	Xδ	Moderately feeble, considerably stronger than Xβ.
2088	452·0	452·6	Xζ	Strong, stronger than Xδ.

The Tmα line was identified by a comparison with Tm obtained from Arendal thorite. The line $\lambda = 539{\cdot}9$ was observed in the spectrum of holmium material, &c., and identified by direct comparison.

The presence of the Diγ and Diδ constituents of didymium, and the absence of other didymium lines is further evidence in support of

the great complexity of didymium. The isolated intensity of the line Xζ is further evidence that Soret's X-earth is a mixture, and this conclusion is also supported by an examination of the spectra which are given, showing the observed relative intensity of the X lines of keilhauite, brevig-thorite, brevig-wöhlerite, and those of an ordinary holmium material. These spectra show that the constituents of what used to be known as holmium present in thorite, wöhlerite and keilhauite are not identical, and the substance, the nitrate of which gives the line Xε in the holmium spectrum, is not present in keilhauite, brevig-thorite, and brevig-wöhlerite. Traces of uranium were observed in the spectrum of keilhauite from Arendal. It is probable that the substances, the nitrates of which give the lines Diδ and Xζ, could be easily obtained from this earth.

120 grams of finely divided gadolinite from Hitterö was evaporated several times with concentrated hydrochloric acid, and the filtered solution precipitated with oxalic acid. The yellow oxide obtained by igniting the oxalates was dissolved in nitric acid, and repeatedly evoporated until no free acid was present. The solution was examined spectroscopically with the following results :—

Observed position of maximum darkness.		Observed wave-length. Krüss and Nilson.	For	Intensity of absorption-bands.
Position of telescope tympanum.	Observed wave-length.			
296	727·8	728·3	Diα	Moderately strong.
398	690·7	690·5	Diκ	Very feeble.
419	683·7	684·0	Tmα	Feeble.
453	673·5	—	?	Very feeble.
525	653·1	654·7	Erα	Moderately feeble, identified by comparison.
574	641·7	640·4	Xα	Feeble.
733	605·0	—	?	Extremely feeble.
788	592·9	591·5	Di	" "
863	580·3	579·2	Diγ	Very strong.
895	575·3	575·4	Diγ	Strong.
1105	542·8	542·6	Xβ	Very feeble.
1128	539·3	539·9	?	Feeble > λ = 539·9.
1241	522·9	523·1	Erβ	Strong.
1257	520·9	521·5	Diδ	Moderately strong.
1347	511·2	512·2	Diε	Feeble.
1589	485·9	585·5	Xδ	Moderately strong.
1728	476·6	477·7	Smβ	Very feeble.
2084	452·2	452·6	Xζ	Very strong.
2203	444·1	444·7	Diι	Barely perceptible.
2453	428·2	428·5	Xη	Very feeble.

The qualitative composition of 12 principal fractions, which were obtained from a mixture of the whole of the gadolinite earths, by fractional decomposition of the nitrates, is given in tabular form,

from the results of spectroscopic examination. This table shows clearly that the nitrates of a whole series of elements exist in all the fractions, and the authors conclude that in order to isolate any particular element from gadolinite, euxenite and other similar minerals which are often employed, at least 20—25 kilos. would be required. The desired result can be most easily attained when the choice of material is guided by a spectroscopic examination.

F. S. K.

Vapour-density and Molecular Weight of Aluminium Chloride. By C. FRIEDEL and J. M. CRAFTS (*Compt. rend.*, **106**, 1764—1770).—Nilson and Petterson's experiments on the vapour-density of aluminium chloride (this vol., p. 788) have led them to the conclusion that the molecule of this compound is represented by the formula $AlCl_3$, and not by the formula Al_2Cl_6, as was deduced from the older experiments of Deville and Troost (*Ann. Chim Phys.* [3], **58**, 257). The authors have previously pointed out that the results obtained by V. Meyer's method are only exact when there is no diffusion of the vapour into the air in the apparatus. At high temperatures, when the volatilisation is very rapid, projection and diffusion must take place to a considerable extent, followed immediately afterwards by condensation in the cool stem of the apparatus. They have therefore redetermined the vapour-density of aluminium chloride by Dumas' method, especial care being taken to obtain the chloride in a perfectly anhydrous condition, and to protect it from moisture during the subsequent operations. The determinations of the vapour-density were preceded by determinations of the vapour-tension.

The aluminium chloride was purified by sublimation, and was obtained in large, colourless crystals, comparatively slightly hygroscopic. Although it volatilises without fusing under ordinary pressure, it melts at 186—187° under a pressure of 2·5 atmos. The results of the determinations of the vapour-tension were as follows:—

Temperature....	167·8°	170·4°	171·9°	175·7°
Vap. tension....	252·1	311·4	316·5	430·7 mm.
Temperature....	182·7°	204·2°	207·5°	213·0°
Vap. tension....	755·4	1793·4	2016·1	2277·5 mm.

The vapour-density was determined at the following temperatures: —218°, 263°, 306°, 357°, 390°, 398°, 400°, 415°, 429°, and 433°. The results obtained agree closely with the value 9·24, calculated from the formula Al_2Cl_6, and confirm the earlier results of Deville and Troost. The lowest temperature at which Nilson and Petterson made a determination was 440°, and the lower values which they obtained indicate that the vapour-density decreases at a high temperature, a result which may be due to the dissociation of the molecule Al_2Cl_6 into the simpler molecules $2AlCl_3$, analogous to the dissociation of iodine, or decomposition may take place in accordance with the equation $Al_2Cl_6 = Al_2Cl_4 + Cl_2$, as in the case of ferric chloride.

The authors' results show that aluminium chloride becomes a perfect gas at 218°, 35° above its boiling point, and the vapour-

density shows no sensible variation between this temperature and 400°. It follows that the molecule of aluminium chloride is correctly represented by the formula Al_2Cl_6. This conclusion it supported by Louise and de Roux's determinations of the vapour-densities of aluminium methide and aluminium ethide. C. H. B.

Hydrochloride of Cobalt Chloride. By P. SABATIER (*Compt. rend.*, **107**, 42).—In presence of increasing quantities of hydrochloric acid, the solubility of cobalt chloride at first decreases and then increases. The following results were obtained at 19°, and are expressed in equivalents of acid and salt respectively, dissolved in 100 equivalents of water.

Hydrochloric acid..	0	1·99	9·16	12·41	16·24
Cobalt chloride	7·77	6·13	3·29	4·57	4·70
Sp. gr.....	1·397	1·307	1·256	1·290	1·314

The last solution evolves hydrogen chloride when exposed to the air, and deposits amethyst-coloured needles of the lower hydrate. The variation in solubility leaves little doubt that the solution contains a hydrochloride, and in fact, when cooled it deposits a pale-blue granular crystalline precipitate which, however, decomposes so rapidly that it could not be analysed. C. H. B.

Explosion of a Tube containing Crystals of Chromous Sulphate. By J. M. VAN BEMMELEN (*Rec. Trav. Chim.*, **6**, 202—204).—A closed tube containing dry crystals of chromous sulphate, ($CrSO_4 + 7H_2O$), kept in a cupboard secured from direct light acquired a greenish tint, and suddenly exploded violently, after having been kept for about a year. No odour of sulphurous anhydride was perceptible on opening the cupboard, and the contents of the tube were syrupy. The explosion was possibly caused by the slow evolution of hydrogen in the tube produced by the decomposition of a trace of water by the salt, and the subsequent decomposition of water of crystallisation liberated in the oxidation of the salt.

The chromous sulphate was prepared by reducing chromic chloride, precipitating with sodium acetate, filtering, washing, and drying the product, the whole operation being conducted in an atmosphere of carbonic anhydride. The crystals had a splendid blue colour. A second tube containing more perfectly dried crystals prepared at the same time has not yet exploded, but the crystals are greenish. N. H. M.

Germanium Oxide. By J. M. VAN BEMMELEN (*Rec. Trav. Chim.*, **6**, 205—208).—Winkler obtained (Abstr., 1886, 985) a gelatinous hydrate of germanium oxide of the formula $GeO + \frac{1}{2}H_2O$ by the action of water on the tetrachloride. The author found the same amount of water in a hydrate of silica obtained under similar conditions (*Arch. Neerland.*, **15**, 330).

When pure germanium oxide is dissolved in water, and evaporated at the ordinary temperature, a hard crust of minute crystals is formed, consisting of the anhydrous oxide; it is not hygroscopic.

Germanium oxide (from the sulphide) when treated with dilute sulphuric acid (1 mol. H_2SO_4 to 4 mols. H_2O) does not dissolve even when heated; the gelatinous oxide, on the other hand, like silicic and stannic acids, retains a certain amount of sulphuric acid.

When a solution of germanium oxide in soda is precipitated with carbonic anhydride, an amorphous substance of an indefinite composition, containing some alkali and water, is formed.

The position of germanium in Mendelejeff and Meyer's tables is in the fourth series between silicium and tin. N. H. M.

Hydrochlorides of Bismuth and Antimony Chlorides. By ENGEL (*Compt. rend.*, **106**, 1797—1800).—The hydrochloride of antimony trichloride is obtained by passing hydrogen chloride into a solution of antimony chloride saturated at 0° until the solution is saturated with the gas, and then cooling it to 0°, when it deposits large crystals of the composition $2SbCl_3,HCl + 2H_2O$, which melt at 16° with evolution of hydrogen chloride; sp. gr. = 2·388.

The hydrochloride of antimony pentachloride, $SbCl_5,5HCl + 10H_2O$, is obtained by adding the pentachloride very gradually to sufficient water to form the hydrate $SbCl_5,4H_2O$; and when the crystals of the latter begin to separate, hydrogen chloride is passed into the liquid, and water is added until the crystals dissolve. The liquid is then cooled to 0°, when it deposits crystals of the hydrochloride which are stable at the ordinary temperatures. The analytical numbers agree more closely with the formula $SbCl_5,5HCl,11H_2O$, but this is not so probable as the formula given.

The hydrochloride of bismuth chloride, $2BiCl_3,HCl + 3H_2O$, is obtained by cooling to 0° a saturated solution of bismuth chloride and hydrochloric acid. It forms crystals which are stable at the ordinary temperature.

All these compounds, like all the hydrochlorides hitherto described, contain water of crystallisation. It is noteworthy that in all the hydrochlorides of chlorides now known, the smallest proportion of water present in the crystals is 2 mols. for each molecule of hydrochloric acid. C. H. B.

Reduction of Gold Chloride by Wood Charcoal. By A. GÖBZ (*Zeit. angewand. Chem.*, **1**, 63—65).—The extraction of gold in the wet way promises to supersede the amalgamation process. Plattner's process consists in treating the ore with chlorine gas and then extracting the soluble chlorides with water. The method appears to require further study to adapt it to the conditions of each particular case. Of the various methods for reducing the ore from the solution so obtained, the latest and most satisfactory is that with wood charcoal. Egleston (*Engineering*, **43**, No. 1116) filters the solution slowly through a bed of powdered wood charcoal, 12 parts of which are found necessary for the reduction of one part of gold. The author prefers to mix the charcoal with the solution and stir. He finds that whereas reduction is not complete even after many days in the cold, it is extremely rapid when the solution is heated. From a solution containing 0·106 gram of gold as chloride in 200 c.c., the gold was

completely precipitated by 1½ hour's boiling with 1 gram of charcoal. From some preliminary determinations of the gases evolved during the reduction, the author concludes that the action is a chemical one, the bye-products of which are carbonic acid, hydrogen, oxygen, and hydrogen chloride, but the results are not very conclusive.

M. J. S.

Atomic Weight of Platinum. By K. Seubert (*Ber.*, **21**, 2179—2187).—A long reply to Dittmar and M'Arthur (*Trans. of Royal Soc. Edin.*, **33**, 561—633), concluding as follows:—"In studying Dittmar and M'Arthur's paper I have received the impression, which is probably also shared by others, that neither in their method of analysis nor in the manner in which their results are calculated have Dittmar and M'Arthur's results that preference which is claimed over mine, and therefore the first two sentences of their conclusions should be altered as follows:—(1) The value Pt = 195·0 (O = 15·96), or Pt = 195·5 (O = 16) obtained by Dittmar and M'Arthur from analyses of potassium platinochloride, is about half a unit too high. (2.) From K. Seubert's experiments, as well as from those of Halberstadt, the mean of 136 analyses gives the value Pt = 194·5, or reduced to a vacuum, Pt = 194·3; this is, at the present time, the most probable value for the atomic weight of platinum."

F. S. K.

Mineralogical Chemistry.

Rare Copper Minerals from Utah. By W. F. Hillberand and H. S. Washington (*Amer. J. Sci.*, **35**, 298—307).—In ore shipments from the Mammoth mine near Denver, Colorado, several rare copper minerals have been found.

1. *Olivenite.*—This mineral occurs in prismatic tabular crystals for which the following axial ratio was obtained: $a : b : c =$ 0·93961 : 1 : 0·672606, a result that differs considerably from that given by Phillips.

2. *Erinite* occurs as a dark green crystalline lining of cavities. Analysis gave the following results:—

CuO.	ZnO.	CaO.	As_2O_5.	P_2O_5.	H_2O.	Fe_2O_3.	Total.
57·67	1·06	0·32	33·53	0·10	7·22	0·14	100·04

The sample contained 3·9 per cent. of insoluble matter not included in the analysis. It lost 0·67 per cent. of water over sulphuric acid, a total of 0·78 at 100°, and 1·14 at 280°, leaving 6·08 per cent. firmly combined.

3. *Tyrolite.*—Some doubt exists regarding the identity of this species. It occurs in scaly masses on quartz. It has a bright apple-

green colour, and a hardness of 2·5. Analysis gave the following results:—

CuO.	ZnO.	CaO.	MgO.	As_2O_5.	P_2O_5.	H_2O.	SO_3.	Total.
46·38	trace	6·69	0·04	26·22	trace	17·57	2·27	99·17

4. *Clinoclasite* occurs distinctly crystallised and in globular forms. It is of a dark bluish-green colour, and has a sp. gr. of 4·38 and a hardness of 2·5 to 3. Analysis of material from a globular mass gave the following results:—

CuO.	ZnO.	As_2O_5.	P_2O_5.	H_2O.	Fe_2O_3.	SiO_2.	Total.
62·44	0·05	29·59	0·05	7·72	0·12	0·06	100·03

5. *Mixite.*—On some specimens of ore was a mineral occurring in delicate tufts of silky needles, which gave on analysis the following results:—

CuO.	ZnO.	CaO.	Bi_2O_3.	As_2O_5.	P_2O_5.	H_2O.	SiO_2.	Fe_2O_3.	Total.
43·89	2·70	0·26	11·18	28·79	0·06	11·04	0·42	0·97	99·31

These results agree fairly well with Schrauf's analysis of mixite.

B. H. B.

Production of Phenacite and Emerald. By P. Hautefeuille and A. Perrey (*Compt. rend.*, **106**, 1800—1803).—A mixture of silica 48 parts, beryllia 15 parts, normal lithium vanadate 200 parts, and lithium carbonate 15 parts, was heated in a platinum crucible at 600—700° for 15 days. The product is a mixture of phenacite, quartz, tridymite, and lithium felspar. The presence of lithium compounds facilitates crystallisation, and promotes the formation of lithium felspar, from which the phenacite itself is formed. The phenacite is isolated by treating the product with water and cold dilute hydrofluoric acid. If the mixture has been heated sufficiently long to decompose the whole of the lithium felspar, all the beryllium is converted into phenacite.

Artificial phenacite is obtained in striated rhombohedral crystals, the angle of which varies from 116° 25′ to 116° 45′, the angle of the natural mineral being 116° 36′. If it has been formed at a high temperature, the dominant form is the prism d' terminated by the rhombohedron p. It is infusible, and resists even the prolonged action of a hot mixture of concentrated sulphuric and hydrofluoric acids. A trace of vanadium imparts to it a greenish colour.

Emerald is obtained from a mixture of silica 12·506 parts, alumina 3·580 parts, beryllia 2·64 parts, and lithium hydrogen molybdate 2 parts. This mixture is heated at a very low red heat for 24 hours, and the temperature is gradually raised to 800°, at which it is maintained for 15 days. At a higher temperature the emerald is completely converted into phenacite. The crystals of emerald can be separated by simple levigation and the yield with the quantities given is about 15 grams. They have the composition SiO_2 67·7; Al_2O_3 19·6; BeO 13·4, which agrees more closely with the calculated numbers than the composition of many natural specimens.

The dominant form is a hexagonal prism *pm*, the height being usually double the diameter of the base; a green colour is given by small quantities of chromic oxide and a yellow colour by ferric oxide. The sp. gr. of some colourless crystals was 2·67. C. H. B.

Nickel Ores from Oregon. By F. W. CLARKE (*Amer. J. Sci.*, **35**, 483—488).—In 1881, extensive deposits of nickel silicates were discovered in Douglas Co., Oregon. The deposits all lie near the surface, in beds from 4 to 30 feet thick. In appearance, the ores are identical with the garnierite and noumeaite of New Caledonia. The silicates vary much in colour and texture, and are unmistakably products of alteration. An analysis of the purest specimen of ore from Riddle, a dark apple-green, compact, and amorphous variety, gave the following results:—

Loss at 110°.	Loss on ignition.	$Al_2O_3 + Fe_2O_3$.	SiO_2.	MgO.	NiO.	Total.
8·87	6·09	1·18	44·73	10·56	27·57	99·90

Of the country rock surrounding the Oregon beds, a large, clean, fresh specimen was analysed (I), and the olivine (II) separated from it was also analysed, the results being as follows:—

	Ignition.	SiO_2.	Al_2O_3.	Cr_2O_3.	Fe_2O_3	FeO.	NiO.	CaO.	MgO.	Total.
I.	4·41	41·43	0·04	0·76	2·52	6·25	0·10	0·55	43·74	99·80
II.	0·57	42·81	—	0·79	2·61	7·20	0·26	—	45·12	99·36

The rock thus contains nickel, and the olivine separated from it contains even a larger proportion. This fact suggests a probable source of derivation for the nickel in the altered beds of ore, and this view is conclusively proved by the microscopic investigation. B. H. B.

Diorite Dyke in Orange Co., New York. By J. F. KEMP (*Amer. J. Sci.*, **35**, 331—332).—Recent workings in the Forest of Dean magnetite mine in Orange Co., New York, prove it to be intersected diagonally by a dyke of diorite, some 6 feet in width. The rock is a typical diorite, dark grey in colour, very fine-grained, and having a sp. gr. of 2·925 to 2·974. On analysis it gave the following results:—

SiO_2.	Al_2O_3.	Fe_2O_3.	CaO.	MgO.	K_2O.	Na_2O.	Loss on ignition.	Total.
48·19	16·79	18·37	6·85	1·32	1·11	5·59	2·31	100·53

The proportion soluble in hydrochloric acid before fusion was 23·5 per cent., of which 18·0 per cent. consisted of ferric oxide. This analysis shows a higher percentage of silica than those of the two dykes described by Hawes from Compton Falls and by Harrington in the neighbourhood of Montreal, also more alumina and iron, but less lime. B. H. B.

Artificial Production of Micas and Scapolite. By DOELTER (*Compt. rend.*, **107**, 42—43).—Natural or artificial silicates of suitable

composition were heated to dull redness with a small quantity of alkaline fluoride. The proper regulation of the temperature is essential. Amongst the minerals used were hornblende, glaucophane, aluminous augite, garnet, chlorite, and andalusite. The micas obtained were biotite, phlogopite, muscovite, and lepidolite, identical with the natural minerals in crystalline form and optical properties, hardness, sp. gr., &c.

In the preparation of muscovite, scapolite was obtained in quadratic crystals identical in properties with those of the natural mineral.

C. H. B.

New Meteorite from California. By G. P. MERRILL (*Amer. J. Sci.*, **35**, 490—491).—This meteorite was found in the San Emigdio Mountains, San Bernardino Co., California. It is of a dull reddish-brown colour, and shows an irregular fracture. It belongs to the chondritic group, and is composed of olivine and enstatite embedded in a base composed of the same minerals in a fragmental and finely divided condition. Nickeliferous iron constitutes 6·21 per cent. of the stone, and occurs in the form of lumps. It is closely associated with pyrrhotite. Very minute colourless crystals of a monoclinic pyroxene also occur. An analysis of the stone gave:—

Metallic portion.	Soluble in HCl.	Insoluble.
6·21	51·26	42·23

The metallic portion yielded—

Iron.	Nickel.	Cobalt.
88·25	11·27	0·48

The soluble portion includes the olivine, iron oxides, and pyrrhotite, whilst the insoluble portion includes the enstatite and pyroxene. The great amount of oxidation which the metallic portion has undergone renders the examination unsatisfactory. B. H. B.

Organic Chemistry.

Constitution of Paracyanogen and Cyamelide. By E. MULDER (*Rec. Trav. Chem.*, **6**, 199—201).—Klason (Abstr., 1886, 1001), showed that when cyanuric iodide, $N<^{Cl\cdot N}_{Cl:N}>CI$, is heated, paracyanogen is formed. The constitution of the latter compound may therefore be represented by two closed rings $N<^{C:N}_{C:N}>C$, attached to each other at two of the carbon-atoms.

In view of the formation of isocyanic and cyanuric acids from cyamelide, the constitution $O<^{C(NH)\cdot O}_{C(NH)\cdot O}>C(NH)$ is suggested for

this compound; the formula is also compatible with the readiness with which free isocyaminic acid polymerises to cyamelide.

N. H. M.

Arsenic Cyanide. By G. W. Blythe (*Chem. News*, **57**, 245).—When arsenious chloride and mercury cyanide are distilled together, an arsenic cyanide appears to be formed. It is a volatile liquid resembling hydrocyanic acid in appearance and odour, and is decomposed by water, probably yielding arsenious and hydrocyanic acids.

D. A. L.

Titanium Nitrocyanide. By C. Reinhardt (*Zeit. angewand. Chem.*, **1**, 124—126).—A specimen of this substance, obtained from the slag of a blast furnace, was purified by heating with hydrochloric, hydrofluoric, chromic, and sulphuric acids. When ignited in air, it gained 29 per cent. The titanium was determined by fusing with potassium hydrogen sulphate, dissolving in cold water, adding acetic acid and sodium acetate, and boiling: 128·72 per cent. of titanic oxide was obtained. Both these results confirm the formula Ti_3CN_4. The compound was not volatile even at a white heat. M. J. S.

Fulminates. By H. W. Warren (*Chem. News*, **57**, 255—256).—Copper fulminate was obtained by digesting an aqueous solution of silver fulminate with copper turnings. It was then reduced by nascent hydrogen by connecting with a small Daniell's cell. In a few hours all the copper was deposited on the negative platinum electrode, and the solution was found to contain hydrocyanic acid and ammonia in large quantities with some ammonium fulminate. In another experiment, copper ammonium fulminate was obtained by adding excess of ammonia to a solution of copper fulminate. The deep-blue crystals obtained in this manner were dried over sulphuric acid and decomposed with hydrogen sulphide, when copper sulphide, carbamide, and ammonium thiocyanate were obtained. When dry silicon fluoride is passed over silver fulminate moistened with petroleum, silver fluoride is formed and an explosive gas escapes. By the action of chlorine, bromine, or iodine, chloropicrin and other allied substances are produced. Experiments on ethyl fulminate are not complete.

D. A. L.

Isolation of the Higher Normal Paraffins from Brown Coal Paraffin. By F. Krafft (*Ber.*, **21**, 2256—2265).—350 grams of crystalline "scale-paraffin," obtained from bituminous schale (?) in the neighbourhood of Halle, were submitted to repeated fractional distillation under 15 mm. pressure. The whole distilled at 185—235° in the first operation, but was finally separated into the following normal paraffins, all of which were crystallised from alcohol in order to remove oily impurities.

Heptadecane, $C_{17}H_{36}$ (5 grams), melting at 22° and boiling at 169—170°; sp. gr. 0·7768 at 22·5°.

Octadecane, $C_{18}H_{38}$, in three fractions boiling at 181—182°:

(1) 3·6 grams melting at 26·5—27·5°; sp. gr. 0·7769 at 28°.

(2) 9 grams melting at 27·2—27·6°; sp. gr. 0·7767 at 28°.

(3) 3·9 grams melting at 27·4—27·6°; sp. gr. 0·7766 at 28°.

Nonadecane, $C_{19}H_{40}$, in three fractions boiling at 193°:
(1) 6·6 grams melting at 31·4—31·8°; sp. gr. 0·7774 at 32°.
(2) 29·8 grams melting at 31·6—31·9°; sp. gr. 0·7775 at 32°.
(3) 25·7 grams melting at 31·6°—32·2°; sp. gr. 0·7775 at 32°.

Eicosane, $C_{20}H_{42}$, in three fractions boiling at 205°:
(1) 19·3 grams melting at 35·5—36·5°; sp. gr. 0·7777 at 36·7°.
(2) 49·4 grams melting at 35·7—36·3°; sp. gr. 0·7777 at 36·7°.
(3) 4·4 grams melting at 36·3—36·6°; sp. gr. 0·7778 at 36·7°.

Heneicosane, $C_{21}H_{44}$, in three fractions boiling at 215°:
(1) 8·8 grams melting at 39·8—40·4°; sp. gr. 0·7784 at 40·4°.
(2) 23·4 grams melting at 39·8—40·2°; sp. gr. 0·7782 at 40·4°.
(3) 5·8 grams melting at 39·5—40·3°; sp. gr. 0·7784 at 40·4°.

Docosane, $C_{22}H_{46}$, in two fractions boiling at 224—225°:
(1) 6·2 grams melting at 43·6—44·1°; sp. gr. 0·7781 at 44·4°.
(2) 10·5 grams melting at 43·6—44·1°; sp. gr. 0·7778 at 44·4°.

Tricosane, $C_{23}H_{48}$ (5·5 grams), boiling at 234° and melting at 47·6—47·8°; sp. gr. 0·7786 at 47·7°.

The total weight of the pure fractions was 263·2 grams.

To prove the identity of these hydrocarbons with the normal paraffins, the various fractions were again rectified or recrystallised from alcohol until their properties were quite constant, and, owing to loss, the total weight of the fractions then only amounted to 216·9 grams.

A second sample of paraffin, sp. gr. 0·7805 at 47·2°, melted at from 42° to 47·2°, distilled between 220° and 270°, and probably consisted chiefly of tricosane and the next two higher homologues, together with decreasing quantities of both higher and lower members of the series.

A third specimen, sp. gr. 0·7804 at 57·5°, melted at 57·5° with previous softening, distilled at 245—295°, and probably contained normal heptacosane ($C_{27}H_{56}$) as chief constituent, somewhat smaller quantities of the two neighbouring homologues, and decreasing quantities of higher and lower members of the series.

All the boiling points given in this paper are for a pressure of 15 mm. F. S. K.

Preparation of Ethyl Nitrite. By W. R. Dunstan and T. S. Dymond (*Pharm. J. Trans.*, **18**, 861—863).—To prepare pure ethyl nitrite the authors dissolve 34·5 grams of pure sodium nitrite in water, make up to 120 c.c., and cool by immersing the vessel in ice and salt. 13·5 c.c. of sulphuric acid is added to a mixture of 32 c.c. ordinary alcohol with an equal volume of water; the liquid is then diluted to 120 c.c., and cooled below 0°. This acid liquid is made to pass gradually through a thistle funnel, with constant stirring, to the bottom of the solution of sodium nitrite in a narrow glass vessel surrounded by ice and salt. At the conclusion of the process a pale-yellow layer of ethyl nitrite is seen floating above the lower layer of sodium sulphate, now semi-solid from the salt crystallising out. The ethyl nitrite thus prepared contains merely traces of alcohol and of water. The former having been removed by agitation with water

in a separating funnel, the latter is eliminated by digestion with anhydrous potassium carbonate.

Thus prepared, the ethyl nitrite is quite pure. It has a sp. gr. of 0·917 to 0·920 at 0° (water at 0° = 1) and boils at 17·5°. It is very unstable in the presence of water, and even of aqueous vapour, especially when acids are also present, although but in minute traces. Hence it can be preserved only when special precautions are taken, as detailed in the paper, and for use in medicine the authors describe the preparation of a 2 per cent. solution in absolute alcohol, with or without addition of glycerol; this solution should be mixed with water only immediately before administration. R. R.

Rhamnose (Isodulcitol). By B. Rayman (*Ber.*, **21**, 2046—2052). —When rhamnose is treated with bromine a *bromide* is obtained which is converted by silver carbonate into a *saccharin*, $C_6H_{10}O_5$. This crystallises in concentrically-grouped, slender needles, melts at 140—142°, is readily soluble in water and alcohol, sparingly soluble in ether. The formation of this compound points to the aldehydic nature of the sugar.

When crystallised rhamnose, $C_6H_{12}O_5 + H_2O$, is boiled with amyl alcohol, the compound $C_5H_{11}O_4{\cdot}CH(OC_5H_{11}){\cdot}OH$ is formed. All alcoholic solutions of rhamnose are lævorotatory: the addition of water destroys this property. (Compare Rayman and Kruis, this vol., p. 667.) N. H. M.

Isodulcitol. By E. Fischer and J. Tafel (*Ber.*, **21**, 2173—2176). —Dextrose, galactose, and isodulcitol are only acted on with extreme slowness by sodium amalgam, whereas levulose and mannose are quickly reduced and converted into mannitol. Isodulcitolcarboxylic acid (this vol., p. 806) is converted into normal heptylic acid when 60 grams of the barium salt are heated for 10 hours with 400 grams of hydriodic acid and 25 grams of amorphous phosphorus. F. S. K.

Compounds of Mannitol. By J. Meunier (*Compt. rend.*, **106**, 1732—1734).—The author has previously described certain compounds of mannitol with aldehydes (this vol., p. 950). In order to obtain the benzoic compound, the hydrochloric acid solution of mannitol (*loc. cit.*) is mixed with benzaldehyde and agitated for a few minutes, when it deposits the compound $C_6H_8O_3(C_7H_6O)_2$. If boiled with dilute sulphuric acid, it assimilates 3 mols. H_2O and splits up into mannitol and benzaldehyde.

The author proposes to call these compounds *mannitoïds*, that just referred to being *tribenzoic mannitoïd*. They are not decomposed by dilute sulphuric acid in the cold, but when boiled with sulphuric acid diluted with four times its volume of water they undergo hydrolysis in the way already indicated.

Trivaleric mannitoïd is obtained in a similar manner, but the solution must contain less hydrochloric acid and less zinc chloride, and must not have become dark coloured. When cooled to a low temperature, the liquid deposits silky needles which are washed with dilute alcohol. They melt at 91°. Trivaleric mannitoïd is not decomposed

by alcoholic potash, but dissolves in sulphuric acid with liberation of valeraldehyde. C. H. B.

Composition of Iodide of Starch. By F. SEYFERT (*Zeit. angewand. Chem.*, **1**, 15—19).—From the fact that the blue compound, precipitated when starch-paste is mixed with a solution of iodine in potassium iodide (and then hydrochloric acid added), requires as much thiosulphate for decoloration as the same quantity of iodine solution without starch, the author infers that the blue compound contains no iodine in the form of hydriodic acid (comp. Mylius, Abstr., 1887, 568). The amount of starch in a solution can be readily estimated from the amount of iodine it precipitates. An excess of standard iodine solution is added, then some hydrochloric acid. The whole is made up to a known volume, and the precipitate allowed to subside. An aliquot portion of the clear liquid is then titrated. 100 parts of starch precipitate 22·79 of iodine, so that, on the assumption that the precipitate contains nothing else, its formula would be $(C_{24}H_{40}O_{20})_6I_7$. M. J. S.

Anhydro-bases of Fatty Diamines. By A. W. HOFMANN (*Ber.*, **21**, 2332—2338).—Diacetylethylenediamine (Mason, Abstr., 1886, 329) is sparingly soluble in ether. It yields a crystalline salt when evaporated at a moderate temperature with hydrochloric acid, but at a higher temperature decomposition sets in. The *platinochloride*, $2C_2H_4(NHAc)_2,H_2PtCl_6$, forms well-defined, deep-red, rhombic crystals, and is very soluble in water. The *aurochloride*, $C_2H_4(NHAc)_2,HAuCl_4$, is a yellow, crystalline compound.

Ethyleneethenyldiamine, $C_2H_4<\begin{matrix}-N\\NH\end{matrix}>CMe$, is obtained when diacetylethyleneamine is distilled or heated above its melting point for a long time, but it is best prepared by heating the diacetyl-derivative in a stream of dry hydrogen chloride. It melts at 88°, boils at 221—224°, and is very soluble in water and alcohol, but almost insoluble in ether. The salts are more stable than those of the acetyl-derivative. The *platinochloride*, $(C_4H_8N_2)_2,H_2PtCl_6$, crystallises in large, pointed prisms, and is very readily soluble in water. The *aurochloride*, $C_4H_8N_2,HAuCl_4$, crystallises in slender needles, and is far less soluble than the platinochloride.

Ethylenebenzenyldiamine, $C_2H_4<\begin{matrix}-N\\NH\end{matrix}>CPh$, is obtained in like manner from dibenzoylethylenediamine. It crystallises from boiling benzene in quadratic prisms, melts at 101°, is very readily soluble in alcohol and ether, less soluble in benzene. The *nitrate*, *hydrochloride*, and *sulphate* are crystalline salts, and readily soluble in alcohol and water. The *platinochloride*, $(C_9H_{10}N_2)_2,H_2PtCl_6$, crystallises in slender needles, and is sparingly soluble in water. The *aurochloride*, $C_9H_{10}N_2,HAuCl_4$, crystallises in needles, and is readily soluble in boiling alcohol, but very sparingly soluble in water.

Trimethyleneethenyldiamine, $C_3H_6<\begin{matrix}-N\\NH\end{matrix}>CMe$, prepared in like manner from diacetyltrimethylenediamine, was obtained in the form

of a brown oil. The *platinochloride*, $(C_5H_{10}N_2)_2,H_2PtCl_6$, forms large, rhombic crystals, and is very readily soluble in water. The more sparingly soluble *aurochloride*, $C_5H_{10}N_2,HAuCl_4$, crystallises in needles.

Trimethylenebenzenyldiamine, $C_3H_6<\begin{matrix}-N\\NH\end{matrix}>CPh$, obtained from dibenzoyltrimethylenediamine, is a crystalline compound which melts under water. It is readily soluble in alcohol and ether, more sparingly in benzene, and insoluble in water. The *hydrochloride*, *nitrate*, and *sulphate* are crystalline, and extremely soluble. The *platinochloride*, $(C_{10}H_{12}N_2)_2,H_2PtCl_6$, and *aurochloride* are sparingly soluble.

Propyleneethenyldiamine, $C_3H_6<\begin{matrix}-N\\NH\end{matrix}>CMe$, is obtained from diacetylpropylenediamine as described above. The *platinochloride*, $(C_5H_{10}N_2)_2,H_2PtCl_6$, forms rhombic crystals, and is extremely soluble in water. The *aurochloride*, $C_5H_{10}N_2,HAuCl_4$, crystallises in small needles. F. S. K.

Hexamethyleneamine-derivatives. By H. E. L. Horton (*Ber.*, **21**, 1999—2002; compare Legler, Abstr., 1886, 327).—*Hexamethyleneamine tetrabromide*, $C_6H_{12}N_4,Br_4$, is produced when bromine is added to an aqueous solution of hexamethyleneamine, or by the action of bromine-vapour on hexamethyleneamine. It is a red, crystalline compound, and is decomposed when boiled with water.

Hexamethyleneamine dibromide is insoluble in ordinary solvents.

Hexamethylene diiodide, $C_6H_{12}N_4I_2$, is obtained when an alcoholic solution of the calculated quantity of iodine is added to an aqueous solution of hexamethyleneamine. It is crystalline, and very sparingly soluble in alcohol.

Hexamethylene tetriodide, $C_6H_{12}N_4,I_4$, prepared similarly, crystallises from acetone in brown, microscopic, rhombic plates. It is readily soluble in acetone, chloroform, and carbon bisulphide, and is decomposed when boiled with water.

Chlorine also produces precipitates in solutions of hexamethyleneamine. F. S. K.

Reaction of Formaldehyde. By J. Plöchl (*Ber.*, **21**, 2117—2119).—When a neutral solution of formaldehyde is treated with ammonium chloride or other ammonium salt at the ordinary temperature, the solution becomes strongly acid. On heating the solution, carbonic anhydride is evolved, and trimethylamine is formed. In a similar manner, mono- and di-methylamine are converted into trimethylamine, whilst pure trimethylamine when heated with a formaldehyde solution remains unchanged. N. H. M.

Action of Hydroxylamine on Acetylacetone. By W. Zedel (*Ber.*, **21**, 2178).—The compound obtained by Combes (*Ann. Chim. Phys.*, 1887, 215) by acting on acetylacetone with hydroxylamine is an anhydride of the constitution $<\begin{matrix}CH\cdot CMe\\CMe:N-\end{matrix}>O$. It is a colourless oil boiling at 141—142°. F. S. K.

Homologues of Diacetyl. By H. v. PECHMANN and R. OTTE (*Ber.*, **21**, 2140—2141). The following homologues of diacetyl have been prepared:—Acetylbutyryl boiling at 128°, acetylisobutyryl boiling at 116°, acetylisovaleryl boiling at 137·5°, acetylisocapronyl boiling at 165°, and acetylcrotonyl. All these compounds are, chemically and physically, analogous to diacetyl. No details are given. F. S. K.

Influence of Negative Groups on the Properties of Compounds. By A. P. N. FRANCHIMONT (*Rec. Trav. Chim.*, **6**, 224—234).—Hydrogen-atoms attached to carbon are not attacked by nitric acid at the ordinary temperature when only one carboxyl-group is attached to the same carbon-atom, as is the case of acetic acid; the presence of a second carboxyl-group renders the compound capable of being attacked, as for instance malonic acid. The fact that acetone is readily acted on shows that it is the carboxyl-group which has the effect of making compounds unable to resist the action of nitric acid, whilst this effect is diminished by the presence of an OH-group attached to carbonyl, but not by methyl. Glycocine, propionic, isobutyric, and succinic acids do not react with nitric acid at the ordinary temperatures, whilst levulinic acid does, owing to the presence of a second carboxyl-group. Hydrogen in ammonia, methylamine, &c., is not attacked by nitric acid, except when a carboxyl-group is attached to the nitrogen; on the other hand, the union of a second carboxyl-group with the nitrogen seems to diminish its effect, diacetamide being less readily attacked than acetamide. Succinimide in which the NH-group forms a chain with the carbon-atoms, does not react with nitric acid. Ethylenecarbamide is also not decomposed by nitric acid. The influence of imidogen seems to be less than that of carboxyl, whilst the simultaneous presence of both groups seems to diminish the effect of the latter, and increase that of the former. In benzene, the six CH-groups so influence each other as to make some of them capable of reacting with nitric acid; the introduction of NO_2 or other negative groups has the effect of making the further action of nitric acid more difficult. The same is observed with nitroethane, which does not yield a dinitro-compound at the ordinary temperature. (Compare Ostwald, *Lehrbuch d. allgem. Chem.*) N. H. M.

Action of Sodium on Ethyl Chloracetate. By R. FITTIG and A. ERLENBACH (*Ber.*, **21**, 2138—2140).—An ethyl salt, $C_8H_{13}ClO_4$, is formed when ethyl chloracetate is gradually added to sodium covered with ether, and the resulting sodium-derivative decomposed with dilute hydrochloric acid. It boils at 157° (45 mm. pressure), cannot be distilled under the ordinary pressure, and is insoluble in water. It yields a *sodium*-derivative, $C_8H_{12}ClO_4Na$, and a *copper*-compound which crystallises from light petroleum in slender, green needles.

A compound, $C_8H_{14}O_4$, is obtained, when the preceding substance is reduced with zinc and acetic acid, in the form of a colourless liquid which boils at 106° (14 mm. pressure), and is rather soluble in water. It yields a *sodium*-derivative, $C_8H_{13}O_4Na$, and a crystalline copper-compound, $(C_8H_3O_4)_2Cu$, which melts at 138°, is readily soluble in

ether, chloroform, and benzene, and when treated with aluminium acetate is converted into a crystalline *aluminium*-compound.

Acetoisopropyl alcohol, $COMe{\cdot}CH_2{\cdot}CHMe{\cdot}OH$, is formed, together with ethyl alcohol and carbonic anhydride, when the preceding compound is boiled with dilute hydrochloric acid. It is a colourless, mobile liquid, boils at 128°, and is miscible with water in all proportions, but is separated from the solution by potassium carbonate. It combines with hydrogen sodium sulphite, forming a crystalline, readily soluble compound, yields a yellowish liquid hydrazide, and combines with hydroxylamine.

When the compound $C_8H_{13}ClO_4$ is left for some time in contact with cold water, it decomposes into alcohol, hydrochloric acid, and a compound, $C_{12}H_{14}O_7$, which is sparingly soluble in water, and crystallises in long, yellow needles melting at 139·5°. The smallest quantity of this substance colours water deep bluish-violet, which changes to yellow when the solution is boiled. It gives an amorphous *barium* salt, $C_{12}H_{12}O_7Ba$, and a crystalline *calcium* salt, $C_{12}H_{12}O_7Ca$, both of which are insoluble in water. F. S. K.

Formation and Decomposition of Ethereal Salts: Action of Acids on Tertiary Amyl Acetate. By D. Konowaloff (*Zeit. physikal. Chem.*, **2**, 6—12).—The action of the haloïd derivatives of acetic acid has been studied. Trichloracetic acid acts rapidly at 99°, but the action becomes less rapid as the temperature decreases, and at 50° is 50 times slower than at 99°. At the higher temperature, 75 per cent. of the acetate is decomposed in six hours, some being converted into trichloracetate. Dichloracetic acid at 99° decomposes 77 per cent. of the acetate in 24 hours, with the formation of some dichloracetate. Monochloracetic acid requires a temperature of 156° to effect an appreciable decomposition, 87 per cent. being then decomposed in two hours. The intention was to have calculated the constants of velocity of these reactions, but owing to the action of the acetic acid set free there is an acceleration as the action proceeds, which interferes with its regular course. The amount of decomposition during the first hour in which this acceleration is at a minimum, if compared with the numbers given by Ostwald for the coefficients of velocity of the above acids, shows, however, that these experiments would give coefficients of velocity of the same order. H. C.

Preparation of Normal Valeric Acid and Dipropylacetic Acid from Ethyl Malonate. By E. Fürth (*Monatsh.*, **9**, 308—322; compare Sedlitzky, this vol., p. 250).—To prepare normal valeric acid the author heats zinc with commercial ethyl malonate and normal propyl iodide in a water-bath for 8—10 hours. The nearly solid products of reaction are repeatedly treated with hydrochloric acid and water, and the ethyl propylmalonic acid dried, fractionated, and converted into barium salt, from which propylmalonic acid is obtained pure by shaking it with hydrochloric acid and ether, allowing the ethereal solution to evaporate, and repeatedly crystallising from benzene. The propylmalonic acid is converted into normal valeric

acid by heating it at 180° in a flask provided with an inverted condenser. The pure acid thus formed boils at 186—186·4°.

Silver normal valerate is best prepared by heating diluted valeric acid with excess of silver oxide. On cooling, the solution deposits the salt in the form of wool-like masses of needles, the solubility coefficient of which is—

$$L = 0·2294 + 0·002668(t - 0·3) + 0·00004543(t - 0·3)^2.$$

Calcium normal valerate crystallises in scales. Its solubility coefficient is—

$$L = 10·238 - 0·07643(t - 0·3) + 0·0006293(t - 0·3)^2.$$

whilst that of the barium salt is given by the formula—

$$L = 21·658 - 0·12348(t - 0·3) + 0·001767(t - 0·3)^2.$$

Dipropylmalonic acid is obtained as a bye-product in the preparation of normal valeric acid as above described. On heating at 180—200° it yields a dipropylacetic acid, identical with that described by Burton (Abstr., 1882, 509). It furnishes a silver salt only slightly soluble in water, an uncrystallisable barium salt, and a calcium salt crystallising in masses of interlaced needles containing 2 mols. H_2O, and having the coefficient of solubility—

$$L = 9·530 - 0·12516(t - 0·3) + 0·0003358(t - 0·3)^2.$$

G. T. M.

Constitution of Ethyl Sodacetoacetate. By A. Michael (*J. pr. Chem.* [2], **37**, 473—530; compare this vol., 674).—Ethyl carbethoxacetoacetate was prepared by the action of ethyl chlorocarbonate on ethyl sodacetoacetate in alcoholic solution, also on the dry salt in dry ether, and on the sodium salt obtained by the action of sodium on a weak ethereal solution of ethyl acetoacetate; in all cases the product is identical, boiling at 127° unchanged (at 17 mm. pressure); it is insoluble in concentrated sodium hydroxide, but is decomposed by a weak solution, after remaining in the cold, into carbonic anhydride and alcohol; it is decomposed by alcoholic sodium ethoxide with formation of carbonic anhydride; by careful treatment in the cold with dry sodium ethoxide, ethyl acetoacetate is formed, but no sodium-derivative of the original salt can be obtained.

Ethyl acetomalonate was prepared, after Lange, by acting on an ethereal solution of ethyl sodiomalonate with acetic chloride; the product was fractionated at 17 mm. pressure, and gave ethyl acetomalonate, boiling at 120°, and ethyl diacetomalonate, boiling at 156°. The latter decomposes when heated at the ordinary pressure, and is a neutral oil dissolving slowly in alkalis, with separation of carbonic anhydride. The former is not completely separable from the latter by its lower boiling point, but can be dissolved by weak alkalis, and precipitated from the solution by mineral acids; its sodium-derivative, $C_9H_3NaO_3$, is easily obtained by the action of alcoholic sodium ethoxide as white prismatic leaves, sparingly soluble in alcohol, insoluble in ether, and freely soluble in water.

The presence of carbonyl in the ether is shown by its behaviour

with hydroxylamine, with which it forms a half-fluid oxime, and phenylhydrazine with which it forms, at 119—121°, white prismatic leaves of the azide, $CH_3{\cdot}CN_2HPh{\cdot}CH(COOEt)_2$.

Ethyl methenyltricarboxylate was obtained after Conrad and Guthzeit (Abstr., 1883, 45) by acting on ethyl sodiomalonate in the cold with ethyl chlorocarbonate. The product was fractionated at 17 mm. pressure, and an oil boiling undecomposed at 137—138° obtained, which solidified on standing, and melted at 28—29°. This substance agrees in physical properties with that obtained by Conrad and Guthzeit, but it differs from it in chemical properties. It dissolves readily in weak alkaline solutions, forming metallic derivatives, and is precipitated unchanged from the solution by mineral acids. If heated with excess of potassium hydroxide, malonic acid, alcohol, and potassium carbonate are formed. With concentrated soda solution, the sodium salt, $C_{10}H_{45}NaO_6$, is obtained in white crystals, soluble in water.

In the author's opinion the only two arguments of any weight against ethyl acetoacetate being a ketone-derivative, are that ethyl bezalacetoacetate does not behave as a ketone, and that several ethyl ketonates resembling ethyl acetoacetate yield true acetyl-derivatives, whereas one cannot be formed from ethyl acetoacetate. He explains away the first objection by supposing that a polymerisation of the benzaldehyde and ethyl acetoacetate first takes place, and that the substance formed then loses water, the ketone character of the ethyl benzalacetoacetate being lost. To meet the second objection the author adduces the formation of acetyl-derivatives from ethyl succinosuccinate, levulinate, and acetophenonecarboxylate; these compounds are undoubtedly ketones, for they all give reactions for carbonyl.

The author supposes that in the formation of ethyl sodacetoacetate from sodium and ethylacetate, an "aldol" polymerisation first takes place, and the sodium acts on the substance so formed.

(1.) $2CH_3{\cdot}COOEt = OH{\cdot}CMe(OEt){\cdot}CH_2{\cdot}COOEt.$

(2.) $OH{\cdot}CMe(OEt){\cdot}CH_2{\cdot}COOEt + Na = ONa{\cdot}CMe(OEt){\cdot}CH_2{\cdot}COOEt + H.$

(3.) $ONa{\cdot}CMe(OEt){\cdot}CH_2{\cdot}COOEt + Na = ONa{\cdot}CMe{:}CH{\cdot}COOEt + EtONa + H.$

This would make ethyl sodacetoacetate a derivative of ethyl β-hydroxycrotonate, which has been already suggested by Geuther.

The carbon in methane will be made more negative by the substitution of a negative radicle for one of the hydrogen-atoms, and the attraction between the negative carbon and positive hydrogen will be weaker than in methane. In the same way the ketone oxygen in ethyl acetoacetate is more negative than in acetone, since the ethyl acetoacetate is formed from acetone by displacing positive hydrogen by the negative carbethoxyl.

Ethyl sodacetoacetate when first formed is $COMe{\cdot}CHNa{\cdot}COOEt$,

but owing to the strongly negative character of the carbonyl oxygen the sodium is attracted to it, and the formula becomes

$$ONa{\cdot}CMe{:}CH{\cdot}COOEt;$$

in this the β-carbon-atom is less negative than the α-carbon-atom, being influenced by the positive sodoxyl and methyl. Thus when an alkyl haloïd, for example, ethyl iodide, acts on ethyl sodacetoacetate, the negative iodine unites with the less negative β-carbon-atom, whilst the ethyl unites with the α-carbon-atom—

$$ONa{\cdot}CMe{:}CH{\cdot}COOEt + EtI = ONa{\cdot}CMeI{\cdot}CHEt{\cdot}COOEt.$$

But this is unstable, owing to the attraction of the sodium for the iodine, and consequently decomposes into sodium iodide and

$$COMe{\cdot}CHEt{\cdot}COOEt.$$

By consideration of the influence of positive and negative radicles on the original condition of the compounds, the author explains various reactions of ethyl sodacetoacetate and its analogues. The rest of the paper consists of applications of this theory to various other classes of organic compounds. A. G. B.

Ethyl Glycidate. By P. Melikoff and N. Zelinsky (*Ber.*, **21**, 2052—2055).—*Ethyl glycidate*, $<^{-O-}_{CH_2}>CH{\cdot}COOEt$, is prepared by gradually adding ethyl iodide to silver glycidate previously mixed with absolute ether, and heating the mixture for some hours on a water-bath. It is a colourless, mobile liquid, of an odour something like that of ethyl malonate, and boils at 161—163° (uncorr.). Sp. gr. = 1·0968 at 21·6°, compared with water at 21·6°, and 1·0933 compared with water at 4°.

Ethyl α-methylglycidate, $<^{-O-}_{CH_2}>CMe{\cdot}COOEt$, is liquid, is insoluble in water, and boils at 162—164°. Sp. gr. = 1·0545 at 15°, and 1·0686 at 0°. Coefficient of expansion = 0·000855.

Ethyl β-methylglycidate, $<^{-O-}_{CHMe}>CH{\cdot}COOEt$, is a liquid of an unpleasant odour, and boils at 172—174°. Sp. gr. = 1·0534 at 15°, and 1·0658 at 0°. The coefficient of expansion = 0·030785.

Ethyl α-β-dimethylglycidate, $<^{-O-}_{CHMe}>CMe{\cdot}COOEt$, boils at 177—178° under 760 mm. pressure. Sp. gr. = 1·0250 at 15°, and 1·0377 at 0°. The coefficient of expansion = 0·000827.

All the above ethyl salts are readily hydrolysed by potash. The alcoholic solutions give no colour reactions with ferric chloride.

Ethyl β-glyceric acid, $OH{\cdot}CHMe{\cdot}CH(OH){\cdot}COOEt$, boils at 225—230°, and is readily soluble in water. N. H. M.

Action of Ethyl Sodiomalonate on Monochloromethyl Ether. By C. Kleber (*Annalen*, **246**, 97—114).—As the preparation of monochloromethyl ether by the exposure of chlorine and methyl ether vapour to diffused sunlight is accompanied by violent explosions, the

author diluted the chlorine with an equal volume of carbonic anhydride. In this way, about 40 grams of the crude product were formed. This was saturated with dimethyl ether, and a continuous current of dimethyl ether vapour and chlorine was passed into the mixture. This method gave a good yield of monochloromethyl ether.

The chief products of the action of ethyl sodiomalonate on monochloromethyl ether are *ethyl methenedimalonate* and *diethyl dimethoxydimethylmalonate*. These substances are separated by adding a solution of sodium in absolute alcohol to the crude product, which causes the sodium compound of ethyl methenedimalonate to be deposited in a crystalline mass. Methenedimalonic acid has already been described by Conrad and Guthzeit as "dicarboxyglutaric acid" (*Annalen*, **222**, 257).

Dimethoxydimethylmalonic acid, $C(CH_2{\cdot}OMe)_2(COOH)_2$, forms triclinic prisms, and is freely soluble in water, alcohol, and ether. The diethylic salt is a colourless oil, boiling between 235° and 243°. The free acid melts at 136—138° when rapidly heated, but if the temperature is slowly raised it gives off carbonic anhydride at 120°, and at a higher temperature methyl alcohol and methoxymethacrylic acid.

W. C. W.

Symmetrical Diethylsuccinic Acids. By C. A. Bischoff and E. Hjelt (*Ber.*, **21**, 2089—2097, 2097—2102, and 2102—2111).—*Ethyl ethylbutenyltricarboxylate* is obtained by the action of ethyl α-bromobutyrate on ethyl sodioethylmalonate, and by the action of ethyl iodide and sodium ethoxide on ethyl butenyltricarboxylate. It boils at 280—282°. Sp. gr. = 1·024 at 22·5° (water at 15° = 1). The *free acid*, $C_9H_{14}O_6$, melts at 150° with evolution of carbonic anhydride and formation of two isomeric diethylsuccinic acids: parasymmetrical diethylsuccinic acid, which is sparingly soluble and melts at 189—190°, and antisymmetrical diethylsuccinic acid which is readily soluble and melts at 129°.

Ethyl butenyltricarboxylate boils at 276° under 754·7 mm. pressure without decomposition; sp. gr. = 1·049 at 25·5° (water at 15° = 1). (Compare Polko, this vol., p. 134.)

Parasymmetrical diethylsuccinic acid (m. p. 189°) crystallises in monoclinic plates. 100 c.c. of water at 23° dissolve 0·61 gram of the acid, whilst 100 parts of water at 95° dissolve 6·7 grams. The acid is readily soluble in acetone, alcohol, ether, and hot glacial acetic acid. The *sodium salt*, $C_8H_{12}O_4Na_2$, is amorphous and dissolves readily in water; the *calcium* with 2 mols. H_2O, *copper* with 1 mol. H_2O, *silver* and *zinc* with 2 mols. H_2O, are also described.

Antisymmetrical diethylsuccinic acid forms rhombic crystals. 100 c.c. of water at 23° dissolve 2·4 grams of the acid which is very soluble in boiling water, in glacial acetic acid, acetone, ether, and alcohol. When quickly heated, it is converted into the anhydride boiling at 245—246°. The *calcium salt* with 1 mol. H_2O is a pulverulent precipitate, the *sodium*, *copper* with 1 mol. H_2O, and *zinc* (with 6 mols. H_2O) *salts* are described. Both acids when heated with resorcinol form fluoresceïn dyes.

Parasymmetrical diethylsuccinic acid undergoes no change when boiled with water for 32 hours. When 4·5 grams of the acid is

heated for two hours at 196°, and the temperature gradually raised during two hours to 218°, 0·14 gram of the anti-acid and 3·8 grams of anhydride are formed. The anti-acid may be reconverted into the para-acid by heating 5 grams of it with 20 c.c. of hydrochloric acid (sp. gr. = 1·1452), or by heating it with water at 190° for 18 hours.

The isomerism of the symmetrical diethylsuccinic acid, which follows that of tartaric and anti-tartaric acids and Reimer's two dibenzyldicarboxylic acids, is discussed. N. H. M.

Determination of the Arrangement of Atoms in Space. Part I. Fumaric and Maleic Acids. By J. Wislicenus (*Annalen*, **246**, 53—96).—The author has pointed out in his treatise "Ueber die raümliche Anordnung der Atome in organischen Moleculen" the existence of two facts which are opposed to the author's theory of the constitution of fumaric and maleic acids, namely, (1) Petri's statement (Abstr., 1879, 373) that fumaric acid is the sole product of the action of bromine on maleic acid in the presence of water, at the ordinary temperature; and (2) the formation of dibromosuccinic acid by the addition of bromine to acetylenedicarboxylic acid as described by Bandrowski (Abstr., 1880, 160).

The action of bromine and water on maleic acid is more complicated than Petri believed. Isodibromosuccinic acid is formed in the first instance, and this acid decomposes, yielding hydrogen bromide and bromofumaric acid. The hydrogen bromide unites with maleic acid, forming monobromosuccinic acid, which afterwards splits up into fumaric and hydrobromic acids.

The chief products of the action of bromine on acetylenedicarboxylic acid in the presence of a small quantity of water are dibromofumaric, monofumaric, and hydrobromic acids. Carbonic and oxalic acids are also formed as well as small quantities of mono- and di-bromomaleic acids. By increasing the proportion of water the yield of dibromomaleic acid is increased, because the action of the hydrobromic acid produced in the primary reaction is weakened by dilution. The proportion of maleic to fumaric acid increases with the temperature at which the maleic acid is decomposed. The temperature must not, however, exceed 230°. W. C. W.

Unsaturated Acids. By G. Pum (*Monatsh.*, **9**, 446—457).—*Ethyl bromomaleate* or *bromofumarate* is formed when ethyl dibromosuccinate (1 mol.) is treated with sodic ethoxide (1 mol.) in alcoholic solution; it is a colourless liquid boiling at 145—155° (30 mm. pressure).

Ethyl acetylenedicarboxylate is obtained when ethyl dibromosuccinate (1 mol.) is treated with an alcoholic solution of sodic ethoxide (2 mols.).

Ethyl dibromomaleate, prepared by brominating the preceding compound, is a colourless, oily liquid boiling at 170—175° (15 mm. pressure) with slight decomposition.

Methyl dibromomaleate is a colourless, oily liquid boiling at 158° (20 mm. pressure).

Hexethyl dimalonylmaleate,

$$CH(COOEt)_2\cdot C(COOEt):C(COOEt)\cdot CH(COOEt)_2,$$

is obtained by treating ethyl dibromomaleate with ethyl sodiomalonate in alcoholic solution. It crystallises in long needles, melts at 75°, boils at 210—212° (15 mm. pressure), and is readily soluble in absolute alcohol, ether, and chloroform, but only sparingly soluble in cold, dilute alcohol.

Dimalonylmaleic acid, $C_{10}H_8O_{12}$, crystallises in small plates, begins to melt at 148° with evolution of carbonic anhydride, and is very readily soluble in water, alcohol, and ether.

The *sodium* salt, $C_{10}H_2O_{12}Na_6 + 10H_2O$, is moderately soluble in water, and crystallises in thick, quadratic plates which lose their water when kept over sulphuric acid or when dried at 120°. The *silver* salt, $C_{10}H_2O_{12}Ag_6$, is crystalline and explodes when heated. Lead acetate, baric chloride, calcic chloride, and copper sulphate produce precipitates in a warm, neutral, aqueous solution of the ammonium salt.

Hexamethyl dimalonylmaleate, $C_{10}H_2O_{12}Me_6$, prepared from methyl dibromomaleate and methyl sodiomalonate, crystallises from alcohol in shining, white needles, melting at 128—130°.

Diglycolylmaleic acid, $C_2(COOH)_2(CH_2\cdot COOH)_2$, is obtained when dimalonylmaleic acid is heated at about 150—160° or evaporated with hydrochloric acid. It crystallises from water in colourless, prismatic forms, melts at 176°, and is readily soluble in ether, alcohol, and water. F. S. K.

Molecular Weight of Citraconic, Itaconic, and Mesaconic Acids, also of Fumaric and Maleic Acids. By E. Paternò and R. Nasini (*Ber.*, **21**, 2156—2158).—From determinations of the molecular weights of citraconic, itaconic, and mesaconic acids by Raoult's method, the authors conclude that these acids are not polymeric, but isomeric, compounds. The results obtained with itaconic and mesaconic acids are in accordance with the molecular formula $C_5H_6O_4$. In dilute solutions of citraconic acid, the depression deviates very considerably from the normal value, probably owing to decomposition of the molecule into anhydride and water, but in concentrated solutions the depression is normal for the molecular formula $C_5H_6O_4$.

Experiments with maleic and fumaric acids show that they are isomeric. The abnormally high results obtained with the latter indicating decomposition into anhydride and water.

In aqueous solutions of dulcitol the values obtained agree with the molecular formula $C_6H_{14}O_6$, it is therefore isomeric with mannitol. Sorbin has the molecular formula $C_6H_{12}O_6$, and is isomeric with glucose. F. S. K.

Formation of Saccharic Acid as a Test for Dextrose in Raffinose and other Carbohydrates: Formation of Furfuraldehyde as a Test for Arabinose. By R. Gans, W. E. Stone, and B. Tollens (*Ber.*, **21**, 2148—2152).—Dextrose, or any carbohydrate from which dextrose can be obtained, yields saccharic acid when

5 grams of the substance is treated with a suitable proportion (generally 1 : 6) of nitric acid, sp. gr. 1·15 (compare this vol., p. 820). This reaction can be conveniently employed for the detection of dextrose, as the saccharic acid is easily identified by analysing the silver salt. Lævulose, galactose, sorbose, and arabinose do not yield saccharic acid by this treatment, but from 5 grams of raffinose silver saccharate can be obtained, and larger quantities (200 grams) yield a crystalline acid which is very similar to, and perhaps identical with, saccharolactone acid. Milk-sugar gives the same acid.

Salep mucus gives saccharic acid and therefore also contains dextrose. Quince mucus contains arabinose but no dextrose.

Pure arabinose does not yield lævulinic acid when treated with sulphuric or hydrochloric acid, but when 5 grams of arabinose are distilled with sulphuric acid of a certain concentration a considerable quantity of furfuraldehyde is obtained, the quantity of which can be estimated by concentrating the distillate, adding ammonia, and weighing the furfuramide produced. Gum arabic, cherry gum, traganth, &c., and other compounds containing arabinose behave similarly. Arabinose yields about 20 per cent., the gums, &c., from 5 to 9 per cent. of furfuramide; 5 grams of dextrose, cane-sugar, galactose, sorbose, and other carbohydrates yield only inappreciable quantities of furfuraldehyde, and the distillate gives a very much less marked reaction with aniline acetate than the distillate obtained from arabinose, &c. Koch's wood-sugar or xylose yields a large amount of furfuraldehyde, but Carragheen moss gives only inappreciable quantities.

Crystalline carbohydrates can be obtained from wheat bran and more especially from the undissolved portions of malt remaining in the manufacture of beer. These carbohydrates, one of which crystallises in needles and has the specific rotatory power $[\alpha]_D = 103°$, are very nearly related to arabinose. F. S. K.

Action of Dilute Mineral Acids on Saccharic Acid. By H. Schrötter (*Monatsh.*, **9**, 442—445).—The author has obtained results similar to those published by Sohst and Tollens (this vol., p. 820), and for the preparation of large quantities of dehydromucic acid employed the following method:—Potassium saccharate is boiled with double its weight of 30 per cent. hydrochloric acid for about three days, the filtered solution mixed with one-third its weight of concentrated hydrochloric acid and again boiled for three days; this operation is repeated as long as any separation takes place, and is generally finished in about nine days. The yield is 16 to 18 per cent. of the acid employed. Only one barium salt was obtained; it crystallised with $3\frac{1}{2}$ mols. H_2O, and lost all its water at 115°. Only one acid was obtained by reducing dehydromucic acid with sodium amalgam; it crystallises in white, anhydrous needles, melts at 148—149°, and its barium salt crystallises with $4\frac{1}{2}$ mols. H_2O. Dehydromucic acid does not react either with acetic oxide or with hydroxylamine. F. S. K.

Ethyl Acetylenetetracarboxylate. By C. A. Bischoff (*Ber.*, **21**, 2085—2089).—Ethyl diethylacetylenetetracarboxylate,

$$CEt(COOEt)_2{\cdot}CEt(COOEt)_2,$$

is obtained by mixing 110 grams of ethyl ethylmalonate with a solution of sodium ethoxide (from 13·4 grams of sodium) and boiling the mixture with 130 grams of ethyl chlorethylmalonate for 40 hours. It is a colourless oil, insoluble in water, and boils at 198—200° under 11·8—4 mm. pressure; sp. gr. = 1·043 at 19° (water at 15° = 1). The salt is not readily saponified by aqueous hydrochloric acid, the reaction being incomplete after eight days' boiling; when the resulting oil is dissolved in cold concentrated sulphuric acid and water added drop by drop, diethylsuccinic acid, melting at 188°, is formed.

When a mixture of 7·5 grams of sodium, 80 grams of alcohol, 52·2 grams of ethyl malonate, and 72·5 grams of ethyl chlorethylmalonate is boiled for 58 hours, the ethyl salts of acetylenetetracarboxylic acid and its diethyl-derivative are formed. N. H. M.

Ethyl Dicarboxyglutarate. By M. Guthzeit and O. Dressel (*Ber.*, **21**, 2233—2235).—Ethyl dicarboxyglutarate (comp. Abstr., 1883, 311) can be easily prepared in large quantity by treating ethyl sodiomalonate with methylene iodide in alcoholic solution. The yield of the pure salt is 70 to 80 per cent. It is a tolerably thick, transparent oil boiling at 200—203°, and is identical with ethyl propanetetracarboxylate (Perkin, Abstr., 1886, 691) and with ethyl methylenedimalonate (Kleber, this vol., p. 97). The free acid melts at 168—170° with evolution of carbonic anhydride and formation of glutaric acid. F. S. K.

Synthesis of Polybasic Fatty Acids. By C. A. Bischoff (*Ber.*, **21**, 2111—2117).—Ethyl propionylpentacarboxylate has a sp. gr. = 1·121 at 15°. The *potassium* and *barium salts* (each with 4 mols. H_2O) were prepared. The *free acid* crystallises from ether in globular groups, and melts at 149—151° with evolution of carbonic anhydride. When the ethyl salt is treated with dry chlorine at 70°, the compound $COOEt{\cdot}CH_2{\cdot}C(COOEt)_2{\cdot}CCl(COOEt)_2$ is formed. When this is treated in the usual manner with ethyl sodiopropionylpentacarboxylate, *ethyl hexaïndecacarboxylate*, $C_6H_4(COOEt)_{10}$, is obtained as a thick, yellowish oil.

Ethyl monochloromalonate reacts with ethyl sodiopropionylpentacarboxylate, yielding *ethyl butonylheptacarboxylate*, which distils at 280—285° under 130 mm. pressure, with partial decomposition. The *chloro-derivative* is a thick, almost colourless oil, sp. gr. 1·169 at 15° (water at 15° = 1).

Ethyl octoïntesserakaidecacarboxylate,

$$C_6H_4(COOEt)_{14} = COOEt{\cdot}CH_2{\cdot}[C(COOEt)_2]_6{\cdot}CH_2{\cdot}COOEt,$$

it obtained by the action of ethyl sodiobutonylheptacarboxylate on the ethyl salt of chlorobutonylheptacarboxylate; it is a thick, nearly colourless oil. N. H. M.

Methylamides and Ethylamides of Trichloracetic and Trimethylacetic Acid and their Action on Nitric Acid. By A. P. N. Franchimont and E. A. Klobbie (*Rec. Trav. Chim.*, **6**, 234—246).—*Trichloracetomethylamide* is prepared by mixing ethyl trichloracetate with a slight excess of an aqueous 30 per cent. solution of methylamine. It crystallises from ether in white crystals melting at 105—106°. Nitric acid slowly acts on it with evolution of gas; no nitro-compound is formed. The ethylamide is also slowly decomposed by nitric acid, with evolution of nitrous oxide.

Trichloracetodimethylamide is obtained by adding drop by drop an ethereal solution of trichloracetic chloride to a well-cooled ethereal solution of dimethylamine, and is purified by distillation in a vacuum. It is a colourless liquid, which solidifies when cooled by means of ether and carbonic anhydride, and is slightly soluble in water. Sp. gr. = 1·441 at 15°. It is not decomposed by nitric acid, even when heated with it.

Trichloracetodiethylamide forms large, transparent crystals, melts at 27°, and is very sparingly soluble in water. It decomposes when distilled in a vacuum (compare Cloëz, Abstr., 1887, 1098). It is not attacked by nitric acid when kept in contact with it for 24 hours. Trimethylacetamide, prepared by heating ammonium trimethylacetate at 220—230°, crystallises from water in long, slender needles, melts at 153—154°, and boils at about 212° under 766·5 mm. pressure. It is at once decomposed by nitric acid at the ordinary temperature with evolution of nitrous oxide.

Methylamine trimethylacetic acid, $(CMe_3{\cdot}COOH)_2NH_2Me$, is formed when trimethylacetic acid and methylamine are heated at 150° for 10—12 hours. It forms slender needles, melts at 81°, boils at 173—175° under 750 mm. pressure, dissolves readily in water, ether, and methyl alcohol, sparingly in light petroleum, and is hygroscopic.

Trimethylacetomethylamide is prepared by the action of trimethylacetic chloride on methylamine both dissolved in ether. It melts at 91°, sublimes at a lower temperature, boils at 203—204° under 759·1 mm. pressure, and is readily soluble in water, alcohol, and ether. It is at once attacked by nitric acid with evolution of nitrous oxide.

Trimethylacetethylamide, prepared in a manner similar to the methylamide, crystallises well, melts at 49°, boils at 203—204° under 765 mm. pressure, and is readily soluble in water, alcohol, and ether. It is decomposed by nitric acid, but not completely in 24 hours.

Trimethylacetodimethylamide is a colourless liquid, boiling at 185—186° under 754 mm. pressure, and is very soluble in water. Sp. gr. = 0·912 at 17°. It does not give off gas when treated with nitric acid at the ordinary temperature, but yields nitrodimethylamine.

Trimethylacetodiethylamide is a colourless liquid, very readily soluble in water. Sp. gr. 0·891 at 15°. It boils at 203°. Neither this substance nor the dimethylamide could be obtained quite pure. It does not evolve gas when treated with nitric acid, but a trace of nitrodiethylamine seems to be formed after 24 hours. N. H. M.

Methylamides and Ethylamides of Heptylic Acid. By A. P. N. Franchimont and E. A. Klobbie (*Rec. Trav. Chim.*, **6**, 247—250).—The compounds are prepared by heating heptylic acid with the amines at 230° for five hours, dissolving the products in ether, and adding potassium carbonate; after 24 hours, water is added, the ethereal solution separated, dried by potassium carbonate, and distilled. They are colourless liquids, slightly soluble in water. The *methylamide* is a thick liquid of a slight odour, boiling at 265·5—266·5° under 758 mm. pressure. Sp. gr. = 0·895 at 15°. The *ethylamide* melts at 5—6°, and boils at 267·5—268·5° under 767 mm. pressure. The *dimethylamide* boils at 242·5—243·5° under 758·5 mm. pressure. Sp. gr. = 0·894 at 15°. The *diethylamide* boils at 257·5—258·5° under 765 mm. pressure. Sp. gr. = 0·881 at 15°.

Both monalkylamides give off gas when treated with nitric acid. The dimethylamide gives off a little gas, nitrodimethylamine being formed. The diethylamide is decomposed by nitric acid with liberation of heptylic acid. N. H. M.

Formation of Amidobutyric Acid by the Action of Ammonia on Crotonic Acid. By Engel (*Compt. rend.*, **106**, 1677—1679).—25 grams of crotonic acid was mixed with 200 c.c. of a solution of ammonia containing 100—115 milligram-molecules in 10 c.c., and the mixture was heated in sealed tubes at 100—105° for 10 hours; the product was then evaporated with lead oxide, and the lead removed from the solution by means of hydrogen sulphide. Amidobutyric acid forms white non-deliquescent crystals, very soluble in water, and soluble in alcohol, from which it is precipitated by ether. Its solution dissolves many metallic oxides; for example, cupric oxide forms a deep blue solution, which yields large crystals of the composition $(C_4H_8NO_2)_2Cu + 4H_2O$. These crystals are only slightly efflorescent, and do not become anhydrous at 100°.

This amidobutyric acid is much more soluble than the α-derivative, and is probably the β-acid, a view which is supported by the fact that its copper salt is also much more soluble than the copper salt of α-amidobutyric acid as described by Heintz. C. H. B.

Amides of Carbonic Acid. By F. Emich (*Monatsh.*, **9**, 378—394).—The following compounds, cyanamide, biuret, amidodicyanic acid, diacyanodiamide, diacyanodiamidine, melamine, ammeline, biuret dicyanamide, melem, ammelide, cyameluric acid, melam, mellon, and mellon hydride are readily decomposed into carbonic anhydride and ammonia with fixation of the elements of water. The decomposition was effected by heating about 0·2 gram of the substance with moderately dilute sulphuric acid; in some cases the sulphuric acid can be replaced by phosphoric acid or alkalis, or by water alone at a sufficiently high temperature.

Compounds such as fulminic acid, fulminuric acid, and isuretin are not decomposed in this simple manner. F. S. K.

Urethane and some of its Derivatives. By E. Mulder (*Rec. Trav. Chim.*, **6**, 169—198).—Urethane dissolved in alcohol is con-

verted by alcoholic potash at the ordinary temperature into potassium isocyanate, NK:CO; no potassium carbamate is formed as intermediate product (compare Arth, *Bull. Soc. Chim.*, **41**, 334). The same reaction takes place when potassium ethoxide is used.

Potassium acts on urethane dissolved in ether with evolution of hydrogen and formation of potassium isocyanate.

Ethyl sodiocarbamate, NHNa·COOEt, obtained by adding sodium to an ethereal solution of urethane, and washing the white precipitate with absolute ether, is very hygroscopic, and dissolves readily in alcohol. The alcoholic solution deposits crystals of sodium isocyanate.

Alcoholic potash, or soda, in presence of water, converts urethane into potassium or sodium carbonate.

Ammonium carbamate, prepared by Basaroff's method, when treated with alcoholic potash, yields a gelatinous compound, probably potassium carbamate, which in presence of a little water is converted into potassium hydrogen carbonate.

When alcoholic solutions of urethane, potash, and mercuric chloride are mixed, the compound NHg·COOEt is obtained as a gelatinous mass; when dried it can be readily powdered, and is nearly colourless. In presence of water, the powder swells up, but without dissolving. A small amount of potassium chloride is formed in the reaction.

Sandmeyer's ethyl chlorimidocarbonate (Abstr., 1886, 611) may be considered as formed by the direct union of normal ethyl cyanate and ethyl hypochlorite. N. H. M.

Reaction Product of Phosphorus Pentasulphide and Carbamide. By C. v. Kutschig (*Monatsh.*, **9**, 406—413).—The compound $C_2H_8N_3PS_2O_2$ is obtained by heating an intimate mixture of carbamide (1 part) and phosphorus pentasulphide (2 parts) at 80—90° for 20 to 30 minutes, and treating the product with water. It crystallises from hot water in shining, monosymmetric crystals, is rather easily soluble in water, sparingly in alcohol, and shows a strongly acid reaction. About one-third of its nitrogen is evolved as nitrogen when it is treated with bromine, and the same quantity is evolved as ammonia when it is boiled with potash. Biuret, and a compound containing both sulphur and phosphorus, are obtained when it is heated with lead oxide. It is decomposed into hydrogen sulphide, ammonia, and phosphoric acid, when heated with moderately concentrated hydrochloric acid, but in absence of air it yields ammonia, hydrogen sulphide, and hypophosphorus acid. A *silver* salt, $C_2H_7AgN_3PS_2O_2$, is obtained when an aqueous solution, acidified with acetic acid, is precipitated with silver acetate. When an aqueous solution of 1 gram of the substance is slowly added to an ammoniacal solution of 2·54 grams of silver nitrate, a white unstable salt, $C_4H_5Ag_3N_3PS_2O_2$, is formed. F. S. K.

Urëids, and their Reactions with Nitric Acid. By A. P. N. Franchimont (*Rec. Trav. Chim.*, **6**, 213—223).—It was previously shown that carbamide as well as its methyl-derivatives are readily

decomposed by nitric acid at the ordinary temperature (*Rec. Trav. Chim.*, **2**, 96 and 330; **3**, 216), generally behaving like the amides and methylamides of the fatty acids. In the present paper, the behaviour of other carbamide-derivatives towards nitric acid is described.

Hydantoic acid, acetylcarbamide, and bromacetylcarbamide, when treated with nitric acid, give off a gas consisting of 1 vol. of carbonic anhydride and 2 vols. of nitrous oxide; biuret gives equal vols. of the two gases, whilst oxaluric acid yields a mixture of carbonic anhydride, nitrous oxide, carbonic oxide, and nitrogen.

Hydantoïn and lactylcarbamide are converted into nitro-derivatives; acetonylcarbamide is not attacked by nitric acid.

Parabanic and dimethylparabanic acids, dimethylmalonureïd and alloxan are not attacked by nitric acid. Barbituric acid and isosuccinylcarbamide yield nitro-derivatives.

The above results show that when the carbamide forms a closed chain with an acid residue, it loses its amidic character, whilst, when an open chain is formed, the compound is at once decomposed by nitric acid. This is confirmed by the behaviour of allantoïn, 1 mol. of which when treated with nitric acid gives off 1 mol. of carbonic anhydride and 2 mols. of nitrous oxide.

Ethylenecarbamide, when treated with nitric acid, yields a nitro-derivative, whilst ethylenedicarbamide is decomposed, yielding equal vols. of carbonic anhydride and nitrous oxide; the disappearance of the amidic character is therefore due to the formation of a closed chain, and not to the presence of carbonyl-groups. Acetylenecarbamide yields a dinitro-derivative.

When ethylidenecarbamide, $CHM<\begin{matrix}NH\\NH\end{matrix}>CO$, is treated with nitric acid, a mixture of equal vols. of carbonic anhydride and nitrous oxide is evolved; this may be due to the splitting up of an unstable nitro-derivative formed as intermediate product with carbamide and a nitro-hydrocarbon. It is, however, mentioned that the constitution of ethylidenecarbamide is not so conclusively established as that of the other compounds examined. Uric acid dissolves in nitric acid with slight development of heat and evolution of carbonic anhydride and nitrous oxide. Caffeïne is attacked slowly, and theobromine still more slowly. Xanthine also gives off gas. N. H. M.

Aspartic Acids. By ENGEL (*Compt. rend.*, **106**, 1734—1737).—The author has previously shown (Abstr., 1887, 917) that fumaric and maleic acids unite directly with ammonia to form aspartic acid. The identity or otherwise of the aspartic acids thus formed is of very great interest in connection with the isomerism of the generating acids. Four varieties of aspartic acid were compared, namely: (A) formed by the combination of fumaric acid with ammonia; (B) formed by the combination of maleic acid with ammonia; (C) the acid obtained by Desaignes' method; and (D) the active acid obtained from asparagin. The combination of fumaric and maleic acids with ammonia takes place at 100° in presence of water. At this temperature aspartic acid is not decomposed by water, and

maleic acid is not transformed into fumaric acid. The molecular weights of maleic and fumaric acid as determined by Raoult's method are identical, and the molecular weights of the four aspartic acids as determined by the same method are also identical. The three acids, A, B, and C have the same degree of solubility which is expressed by the equation $\gamma = 517 + 21{\cdot}693t - 0{\cdot}165t^2 + 0{\cdot}0079t^3$. The solubility of the active acid is different, the equation in this case being $\gamma = 372 + 14{\cdot}1t - 0{\cdot}18124t^2 + 0{\cdot}0053t^3$. Cupric aspartate prepared from the four acids always crystallises with 9 mols. H_2O, but the crystals from the active acid D are pale-blue needles, whereas the crystals from the three acids A, B, C, whilst identical among themselves, are darker and less definite than those from the acid D.

When the active acid is exposed to the air, it soon becomes covered with mould, and if some of this mould is transferred to solutions of the other three acids, they soon acquire a lævorotatory power. Advantage is taken of the insolubility of the active aspartic acid in a saturated solution of the inactive acid to isolate the active acids formed by the action of the mould, and when the new active acids are mixed with an equal weight of the active acid from asparagin, they produce an inactive acid identical with the three acids, A, B, and C.

From these results it follows that fumaric and maleic acids combine with ammonia to form the same inactive aspartic acid.

C. H. B.

Ethyl Telluride. By A. Marquardt and A. Michaelis (*Ber.*, **21**, 2042—2046).—*Triethyl telluride chloride*, $TeEt_3Cl$, is obtained by adding an ethereal solution of tellurium tetrachloride, drop by drop, to a similar solution of zinc ethide; it crystallises from alcohol in colourless plates, melts at 174°, dissolves readily in alcohol, sparingly in ether. It deliquesces when exposed to air; when treated with moist silver oxide, the hydroxy-derivative is formed; this yields with hydriodic acid Becker and Cahours' iodide, $TeEt_3I$ (*Ann. Chim. Phys.* [5], **10**, 50), melting at 90—92°. When the triethyl-compound is heated with an excess of zinc ethide at 100—110°, butane and diethyl telluride are formed. The latter is obtained as a reddish oil boiling at 140°; the pure diethyl telluride from potassium telluride boils at 137—138°, not 98° (Wöhler, *Annalen*, **35**, 111, and Heeren, *Jahresber.*, 1861, 565).

Triethyl telluride bromide is a white, deliquescent substance, melts at 162°, dissolves readily in water and alcohol, and is insoluble in ether.

N. H. M.

Alkyl-derivatives of Bismuth. By A. Marquardt (*Ber.*, **21**, 2035—2042; compare Abstr., 1887, 802).—Hydrogen sulphide reacts with methylbismuthine oxide and with dimethylbismuthine oxide in alcoholic solution, with formation of voluminous orange-coloured precipitates readily soluble in ammonium sulphide. The compounds could not be purified and are probably represented by the formulæ BiMeS and Bi_2Me_4S. When heated, bismuth sulphide is formed.

Triisobutylbismuthine, $Bi(C_4H_9)_3$, prepared in a manner similar to

the trimethyl-compound (*loc. cit.*) by adding bismuth bromide to zinc isobutyl, forms a heavy, colourless liquid having a slight odour of butyl-compounds; it fumes in air and burns on filter-paper with a dark yellow flame. It boils at 160—162° under 74 mm. pressure with partial decomposition.

Diisobutylbismuthine bromide, $BiBr(C_4H_9)_2$, obtained by adding bromine to a solution of the tributyl-compound in light petroleum, forms white, tabular crystals readily soluble in alcohol. It burns when exposed to air.

Isobutylbismuthine dibromide, $BiBr_2{\cdot}C_4H_9$, is formed when isobutylbismuthine and bismuth bromide in molecular proportion are dissolved together in dry ether. It melts at 124°, crystallises in honey-coloured prisms readily soluble in alcohol and glacial acetic acid, rather sparingly in ether; when exposed to air, it remains unchanged.

Diisobutylbismuthine hydroxide is a yellow, crystalline compound which inflames when exposed to air.

Triisoamylbismuthine, $Bi(C_5H_{11})_3$, is a colourless, slightly fuming liquid which boils at 190—200° under 70 mm. pressure with partial decomposition.

Diisoamylbismuthine bromide forms white crystals readily soluble in alcohol, less so in ether; it inflames in air.

Isoamylbismuthine dibromide is obtained by mixing ethereal solutions of bismuth bromide and triisoamylbismuthine, and on evaporating the ether separates as a honey-yellow oil, which solidifies to a pulverulent mass when cooled; it is insoluble in light petroleum, soluble in alcohol and ether. No halogen compounds of the alkyl-derivatives of bismuth could be obtained. N. H. M.

Condensation of Glyoxal with Ethylmalonate and Acetoacetate. By M. Polonowsky (*Annalen*, **246**, 1—32).—*Ethyl dihydroxybutanetetracarboxylate*, $CH(COOEt){\cdot}CH(OH){\cdot}CH(OH){\cdot}CH(COOEt)_2$, is obtained in the form of a thick syrup by the action of a concentrated aqueous solution of zinc chloride on a mixture of glyoxal (1 mol.) and ethyl malonate (2 mols.). When a mixture of glyoxal and ethyl acetoacetate is treated with a concentrated aqueous solution of zinc chloride at the ordinary temperature, a syrupy liquid is produced which is extracted with ether. The product is separated into two distinct portions by treatment with sodium hydroxide. The portion soluble in alkali contains *methylfurfurancarboxyacetic* or "*sylvanecarboxyacetic*" acid, $O<\begin{matrix}C(CH_2{\cdot}COOH){:}CH\\ CMe{=}{=}C(COOH)\end{matrix}>$. This acid is best prepared by hydrolysis of the crude product at the ordinary temperature. After three or four days, the alkaline solution is acidified; the acid is then deposited in crystals which are purified by recrystallisation from alcohol and afterwards from hot water. The pure acid melts at 207°, decomposes at 250°, crystallises in needles, and dissolves freely in alcohol and also in alkalis and alkaline carbonates. The *ammonium salt*, $C_8H_6O_5(NH_4)_2 + \frac{1}{2}H_2O$, is deposited in microscopic needles on passing ammonia gas into an alcoholic solution of the acid. The *barium* salt, $C_8H_6O_5Ba + 2H_2O$, forms transparent needles. The

silver salt contains 1 mol. H_2O. It is much more soluble in hot than in cold water.

When an alcoholic solution of the acid is saturated with hydrogen chloride, the normal and the acid ethereal salts are formed. The mixture of the two methyl salts is separated by treatment with a solution of sodium carbonate, in which the normal ether is insoluble and the acid salt soluble. $C_6H_6O(COOMe)_2$ is an uncrystallisable liquid. The acid salt, $COOH{\cdot}C_6H_6O{\cdot}COOMe$, crystallises in needles and melts at 98°. It yields crystalline barium and silver salts. The latter has the formula $COOMe{\cdot}C_6H_6O{\cdot}COOAg$. The acid ethylic salt, $COOH{\cdot}C_6H_6O{\cdot}COOEt$, melts at 76°. The dicarboxylic acid is converted into *sylvanacetic* acid, $C_4OHMe{\cdot}CH_2{\cdot}COOH$, by exposure to a temperature somewhat above its melting point. The new acid melts at 137—138°. It dissolves freely in alcohol and is volatile in a current of steam. The silver salt, $C_7H_7O_3Ag + \frac{1}{2}H_2O$, is slightly soluble in hot water. The barium salt crystallises with $4\frac{1}{2}$ mols. H_2O.

That portion of the original condensation product of glyoxal and ethyl acetoacetate which is insoluble in alkalis, separates in the course of time into a thick oil and a small quantity of a crystalline compound, both having the composition $C_{14}H_{18}O_6$. The crystals melt at 139° and are sparingly soluble in the ordinary solvents. The oil consists of diethylsylvanecarboxyacetoacetate, $COOEt{\cdot}C_4OHMe{\cdot}CHAc{\cdot}COOEt$. It is miscible with alcohol, ether, benzene, and chloroform.

Sylvanecarboxyacetic acid is converted into acetonylacetone on heating it at 200° with water containing a minute quantity of hydrochloric acid. W. C. W.

Thiophen. By B. Pawlewski (*Ber.*, **21**, 2141—2142).—From determinations of its vapour-density at various temperatures, the author concludes that thiophen is not decomposed when heated at 336—340°. The critical temperature was found to be 317·3°, and the critical pressure 47·7 atmospheres, from which Van der Waal's constants are calculated to be $a = 0{\cdot}04145$, $t = 0{\cdot}00566$, and $v = 0{\cdot}01698$. (Compare R. Schiff, Abstr., 1885, 971.)

F. S. K.

Constants of Benzene. By B. Lachowicz (*Ber.*, **21**, 2206—2210.)—The discordant results obtained by many experimenters in the determinations of the constants of benzene, are probably due to the air which is dissolved by benzene in considerable quantities, and is very difficult to get rid of. The author found the boiling point to be 80·39° at 760 mm. pressure and the melting point 5·42°. Some benzene melts at 4·84° because of the air which it contains; benzene containing air can be cooled to −4·2° before it solidifies, whereas benzene free from air cannot be cooled below +3° or sometimes even +5° without solidifying. The sp. gr., reduced to 4° and a vacuum, is 0·87270 at 24·27°, whereas benzene which is not freed from air has a sp. gr. 0·87451 at the same temperature.

In the following table the mean coefficient of expansion for the temperature $t°$ is given in the column α. Column v gives the volume calculated from the coefficients of expansion, Kopp's value for 5°

being taken as the starting point. The column $\left(\frac{g}{4^\circ}\right)$ gives the sp. gr. calculated from the sp. gr. at 24·27° reduced to 4° and a vacuum. Column $\left(\frac{1}{g}\right)$ gives the specific and column $\left(\frac{m}{g}\right)$ the molecular volume (C = 11·97).

t°.	α.	t°.	v.	$\frac{g}{4^\circ}$.	$\frac{1}{g}$.	$\frac{m}{g}$.
—	—	0	1·00000	0·89408	1·11846	87·04
5— 6	0·0012185	5	00589	88885	12505	87·53
6—10	11319	6	00712	88776	12641	87·66
10—15	11561	10	01168	88376	13153	88·06
15—20	11634	15	01752	87868	13807	88·56
20—25	12097	20	02344	87360	14468	89·08
25—30	12371	25	02468	87255	14607	89·19
30—35	12384	30	03102	86718	15316	89·74
35—40	12512	35	03740	86184	16030	90·29
40—45	12749	40	04389	85649	16756	90·86
45—50	13428	45	05055	85106	17502	91·44
50—55	13599	50	05760	84539	18289	92·05
55—60	12417	55	06479	83968	19093	92·68
60—65	13433	60	07140	83450	19833	93·25
65—70	13455	65	07860	82893	20673	93·88
70—75	13469	70	08586	82339	21449	94·51
75—84	13429	75	09317	81784	22273	95·15
—	—	80	10051	81239	23094	95·79
—	—	80·39	—	81196	23159	95·84

F. S. K.

Physical Constants of Benzene. By A. Horstmann (*Ber.*, **21**, 2211—2222).—The author discusses the various results which have been obtained in determinations of the heat of combustion of various saturated and unsaturated hydrocarbons, including benzene, and concludes that in physical properties benzene is intermediate between the saturated and unsaturated compounds, a conclusion which Baeyer has already drawn from a study of the chemical properties.

F. S. K.

Constitution of Benzene. By A. v. Baeyer (*Annalen*, **245**, 103—190; compare Trans., 1887, 208).—The reduction products of terephthalic acid have no longer the character of benzene-derivatives, the di- and tetra-hydro-acids behaving like unsaturated, whilst the hexahydro-acid behaves like a saturated fatty acid. The latter acid yields a mono- and a di-bromo-derivative in which the bromine atoms taken up the α-positions: $COOH\cdot CBr<\begin{matrix}CH_2\cdot CH_2\\CH_2\cdot CH_2\end{matrix}>CH\cdot COOH$ and $COOH\cdot CBr<\begin{matrix}CH_2\cdot CH_2\\CH_2\cdot CH_2\end{matrix}>CBr\cdot COOH$. The derivative obtained by the addition of hydrogen bromide to tetrahydroterephthalic acid has the constitution [Br = 2].

Tetrahydroterephthalic acid has the constitution

$$COOH\cdot C\begin{matrix}CH\cdot CH_2\\ CH_2\cdot CH_2\end{matrix}>CH\cdot COOH,$$

which is proved as follows: (1.) The acid combines with 1 mol. of bromine, yielding a saturated acid not identical with the two known dibromohexahydro-acids in which the bromine is in the para-position; when reduced, the bromine is displaced by hydrogen. (2.) In the hydrobromide of the tetrahydro-acid, the bromine is in the β-position, as two acids different from it in which the bromine are in the α-position are formed by the bromination of the hexahydro-acid, and a third is not conceivable. (3.) The formation from the dibromide of the tetrahydro-acid of a hydroxy-acid which yields tetrabromopyrocatechol when treated with bromine.

Tetrahydroterephthalic acid dibromide, when boiled with aqueous soda, yields the same dihydro-acid as that obtained by reduction. The dihydro-acid combines with 2 mols. of bromine, yielding a saturated acid from which the same dihydro-acid can be regenerated by heating it with an alkali. It also yields a dibromide. This, when treated with bromine, yields a lactone ether which is reduced by zinc-dust and acetic acid to the hydrogen alkyl salt of the dihydro-acid, and is therefore derived from a tetrabromide in which two pairs of bromine-atoms are attached to adjacent carbon-atoms. The constitution of the dibromide of the tetrahydro-acid and of the dihydro-acid is expressed by the formulæ $COOH\cdot CBr<\begin{matrix}CH_2\cdot CH_2\\ CHBr\cdot CH_2\end{matrix}>CH\cdot COOH$ and $COOH\cdot C\begin{matrix}CH\cdot CH_2\\ CH:CH\end{matrix}>CH\cdot COOH$.

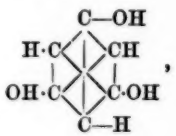
The dibromides of tetra- and di-hydroterephthalic acids both give up 2 mols. HBr when treated with alkalis; the former reaction is readily explained by the production of two double unions, whilst the adoption of this explanation in the second case would involve the assumption that in the elimination of 1 mol. of HBr a monobromide of the constitution

$$[COOH : H : H_2 : H\cdot COOH : Br : H = 1:2:3:4:5:6 \text{ or } 1:2:3:4:6:5 \text{ or } 1:5:3:4:2:6]$$

is formed. The bromine-atoms must therefore be eliminated along with hydrogen-atoms from the ortho-para- or from the meta-meta-positions. For this reason, the author concludes that the constitution of terephthalic acid cannot be explained by any theory hitherto suggested; this view is supported by a comparison of the stability on the one hand of terephthalic acid, and on the other its di- and tetra-hydro-derivatives. Whereas the latter have the properties of unsaturated fatty acids, terephthalic acid is exceedingly stable, and it follows that terephthalic acid cannot in the ordinary sense contain double combinations. Claus' diagonal formula would give a satisfactory explanation of the behaviour of terephthalic acid, but must be rejected as not harmonising with the constitution of dihydro-terephthalic acid.

The formula for benzene which the author now adopts is that first suggested by Kekulé (*Annalen*, **137**, 158), but afterwards given up

owing to the difficulty of explaining the trivalency of carbon which it involved, a difficulty now got over by the representation of atoms in space. If it is assumed that the six carbon-atoms of benzene, in consequence of the attraction of free affinities, turn on the axes (formed by the sides of the hexagon) in such a way that their direction tends inwards towards the plane of the ring, the six points of attack lie symmetrically within the ring, and may paralyse one another, so as to be practically unavailable, or in other words, carbon in benzene is trivalent. A figure is given showing the relative position of the carbon- and hydrogen-atoms. The carbon-atoms are represented by equidistant points, and the three bonds of each carbon-atom by three lines forming three angles of 120°, so that the inner bonds meet to one point in the middle. The fourth valency forms nearly a right angle to the plane of the drawing. It is probable that all the hydrogen-atoms are on one side of the plane of the ring, as when mellitic acid is reduced a maleïnoïd hexahydro-acid is formed: all the carboxyl-groups are therefore on the same side, and hence also all the hydrogen-atoms of benzene. In the model of benzene, two groups of opposing forces are represented, the central valencies tending to draw the atoms towards the middle point, and the peripheral bonds striving to widen out the ring. In benzene these forces are in equilibrium, but as soon as the two central valencies become (by means of reducing agents) united to hydrogen, the equilibrium is destroyed, and the tension of the peripheral bonds (now in excess), expanding the ring, causes the four remaining valencies to associate to double unions. The best example of this is phloroglucinol—



which when treated with hydroxylamine yields a derivative of hexamethylene.

The geometrical isomerism of hexahydroterephthalic acid depends on the presence of asymmetric carbon-atoms. This isomerism is, however, to be distinguished from that which depends on the presence of two asymmetric carbon-atoms in open chains, as in the case of tartaric acid. Whilst this acid retains its geometrical isomerism when one of the carbon-atoms loses its asymmetric character by the displacement of hydroxyl by hydrogen, this is not the case with hexahydroterephthalic acid. On displacing one of the carboxyl-groups of the hexahydro-acid by hydrogen, hexahydrobenzoic acid is formed which no longer contains asymmetric carbon and also no longer shows geometrical isomerism. The isomerism of hexahydroterephthalic acids corresponds exactly with that of fumaric and maleïc acids. The one acid (corresponding with fumaric acid) is sparingly soluble in water, crystallises in prisms and sublimes when heated. The other forms large crystals, dissolves readily in water, and melts at 160°. It may therefore be safely assumed that in the readily soluble

hexahydro-acid both carboxyl-groups are situated on the same side of the plane of the ring.

Terephthalic acid is best prepared by gradually adding dry bromine (2 mols.) to 100 grams of paraxylene contained in a reflux apparatus and heated at 150°. The product is digested in a water-bath with an alcoholic solution of potassium acetate. The acetate is heated on a water-bath with 1 litre of water, 500 grams of aqueous soda (sp. gr. = 1·22), and treated gradually with 4·5 litres of 10 per cent. permanganate solution, more being afterwards added, if necessary. After being heated for three hours, it is filtered, treated with alkali, the solution heated in a water-bath and gradually acidified. In this way, the terephthalic acid is made to separate in needles. The methyl salt dissolves in 300 parts of hot water, and is sparingly soluble in methyl alcohol, more readily in ethyl alcohol. It forms rhombic plates (brachypinakoïd and primary pyramid); $a:b:c = 0{\cdot}84281 : 1 : 3{\cdot}0826$. Paraxylene forms monoclinic crystals: $a:b:c = 2{\cdot}32 : 1 : 2{\cdot}34$; $\beta = 69\frac{1}{2}°$.

Dihydroterephthalic acid (contrary to a previous statement, *loc. cit.*) is readily obtained by dissolving 50 grams of terephthalic acid in a mixture of 120 grams of aqueous soda (sp. gr. = 1·22), and 400 c.c. of water, and adding 3·5 kilos. of 3 per cent. sodium amalgam and sufficient water to prevent the separation of sodium salts. A small amount of paratoluylic acid is formed, to get rid of which the crude acids are converted into ethyl salts which are easily separated from each other. It resembles terephthalic acid in appearance as well as in solubility. It does not melt, but sublimes with partial decomposition into terephthalic acid. The *methyl salt* prepared similarly to methyl terephthalate, crystallises in monoclinic plates ($a:b:c = 1{\cdot}52 : 1 : 2{\cdot}79$; β = about 74°), dissolves in hot alcohol, less in hot water, and melts at 130°. When the methyl salt is heated with alcoholic potash, an intense yellow coloration is produced. The salt is less readily saponified than that of terephthalic acid; on treating it with alcoholic potash and then with acid, an ether acid is formed which melts at 225°.

In determining the behaviour of substances towards permanganate solution, it is best to use sodium carbonate instead of soda, as then no manganate is formed in the conversion of the permanganate into the brown oxide. Terephthalic acid is not changed in the cold by permanganate and only very slowly when warmed. Di- and tetra-hydroterephthalic acids are immediately oxidised in the cold; the hexahydro-acid is not oxidised in the cold but rather quickly when warmed. The halogen-derivatives behave similarly. The method is useful to separate all unsaturated from saturated acids. The results obtained with a number of acids are given.

Dihydroterephthalic acid dihydrobromide, $C_8H_{10}Br_2O_4$, is prepared by heating methyl dihydroterephthalate with aqueous hydrogen bromide (saturated at 0°) for three hours at 120°. It is a crystalline powder. When treated with silver oxide, carbonic anhydride is evolved and an acid—probably dihydrobenzoic acid—melting at about 115° is formed. The *methyl salt*, $C_{10}H_{14}Br_2O_4$, crystallises in thin monoclinic plates; $a:b:c = 1{\cdot}5477 : 1 : 3{\cdot}0027$; $\beta = 85°\ 17'$. It melts at 166°. It is much more stable than the acid, and is scarcely changed when heated

with acetic acid and silver acetate at 130°; at 160°, ethyl dihydrophthalate is formed. When reduced, both the acid and the salt yield the hexahydro-derivative.

Methyl dihydroterephthalate dibromide, $C_{10}H_{12}Br_2O_4$, is obtained by adding 35 grams of bromine dissolved in 100 grams of chloroform in three portions to a solution of 20 grams of methyl dihydroterephthalate in 200 grams of chloroform, taking care to avoid too great rise of temperature. After a quarter of an hour the solution is cooled down, decolorised with sulphurous acid, washed with potassium carbonate solution, and dried with potassium carbonate. The crude salt, which contains the methyl dihydroterephthalate or terephthalate, is saponified with a mixture of sulphuric and acetic acids; the *methyl hydrogen salt* thus obtained forms colourless plates. This is reconverted into the dimethyl salt in the usual manner. It melts at 90°. When reduced with glacial acetic acid and zinc-dust, it yields ethyl dihydroterephthalate; cold alcoholic potash converts it into ethyl hydrogen terephthalate. The *free acid* obtained by heating the methyl salt with hydrobromic acid on a water-bath for 8 to 10 hours, crystallises from hot water in small rhombohedra. When methyl dihydroterephthalate dibromide is treated with an excess of bromine, it is converted into the *tribromolactone ether* of *hexahydroterephthalic acid*,

$$COOMe\cdot C_6H_6Br,<\overset{CO}{\underset{O}{}}>$$ [COOMe : Br_3 : CO,O = 1 : 3 : 4 : 5 : 4, 6].

This forms colourless plates, melts at 187—188°, dissolves sparingly in ether, more readily in methyl and ethyl alcohols, and still more in warm chloroform and glacial acetic acid. When the lactone ether is reduced, hydrogen methyl dihydroterephthalate is formed. It is probable that in the preparation of the lactone the tetrabromide of methyl dihydroterephthalate, [Br·COOMe : $(HBr)_3$: H·COOMe : H_2 = 1 : 2 : 3 : 6 : 4 : 5], is formed as intermediate product. The above experiments show that dihydroterephthalic acid contains two ethylene-like combinations. Tetrahydroterephthalic acid (Abstr., 1887, 370) yields sparingly soluble *barium* and *potassium salts*, the former crystallising in characteristic triclinic plates. The methyl salt forms long, lustrous needles, like caffeïne, and dissolves very readily in most solvents; it has no odour and shows no fluorescence when pure. The rose-coloured precipitate previously obtained with sodium ethoxide is also due to an impurity. The tetrahydro-acid unites with bromine and with hydrogen bromide more readily than the dihydro-acid. The dibromide, $C_8H_{10}Br_2O_4$, is best obtained by adding bromine to the solution of the acid in phosphorus pentachloride, decolorising the product with sulphurous acid, and recrystallising it from water. It dissolves sparingly in water, readily in ether. When boiled with aqueous potash, it yields the dihydro-acid, and when reduced with zinc-dust and acetic acid it is converted into the tetrahydro-acid. The experiments previously made (*loc. cit.*) to determine the position of the bromine-atoms were repeated and confirmed. The methyl salt of the dibromide is prepared from the methyl salt of the tetrahydro-acid; the product contains crystals of two forms and consists probably of two geometric isomerides; the larger crystals (which are in excess)

melt at 81°. When the methyl salt is reduced, or is heated with alcoholic potash, the tetrahydro-acid is formed.

Tetrahydroterephthalic acid hydrobromide,

$$COOH\cdot CH<\begin{matrix}CHBr\cdot CH_2\\ -CH_2\cdot CH_2-\end{matrix}>CH\cdot COOH,$$

is prepared by heating 1 gram of the tetrahydro-acid with 7·5 c.c. of hydrobromic acid (saturated at 0°) for one day at 100° and one day at 125°. To remove traces of unchanged tetrahydro-acid, the product is treated with permanganate. It has about the same solubility in water as the tetrahydro-acid, but is much more readily soluble in ether. It is converted by aqueous or alcoholic potash into the tetrahydro-acid and by zinc-dust and glacial acetic acid into the hexahydro-acid. When warmed with water and silver oxide, an acid is obtained, probably tetrahydrobenzoic acid. *Methyl tetrahydroterephthalate hydrobromide* forms monoclinic crystals; $a : b : c = 2·9050 : 1 : 1·3694$; melts at 94—95°, and is sparingly soluble in ether and light petroleum. When reduced, the methyl salt of fumaroïd hexahydroterephthalic acid is formed.

In the preparation of the above hydrobromide, a considerable amount of an acid readily soluble in water is formed. This contains bromine, and goes over into the tetrahydro-acid on distilling off the hydrobromic acid. It is isolated by nearly neutralising the acid solution with sodium carbonate, saturating with sodium chloride, and extracting with ether. It forms a syrup which afterwards becomes crystalline. The acid seems to be a geometrical isomeride of the hydrobromide just described, derived from maleïnoïd hexahydroterephthalic acid, as when treated with zinc-dust and glacial acetic acid, it yields the cis-hexahydro-acid, whilst the ordinary hydrobromide yields scarcely anything but the ordinary hexahydro-acid.

Hexahydroterephthalic acid (fumaroïd acid) is best prepared from tetrahydroterephthalic acid dibromide. It crystallises from water in groups of prisms, like ammonium chloride, dissolves readily in alcohol and acetone, sparingly in ether, in 75 parts of boiling water, and in 1162 parts of water at 16·5°. The *alkali* and *barium salts* are readily soluble; the *calcium salt* forms sparingly soluble quartz-like crystals; the *copper*, *lead*, and *silver salts* are sparingly soluble. The methyl salt forms triclinic plates or prisms, having a slight odour of ethyl malonate, soluble in 200 parts of hot water.

Cis-hexahydroterephthalic acid (maleïnoïd acid) is best obtained by reducing the readily soluble hydrobromide of the tetrahydro-acid, and can also be prepared from the dibromohexahydro acid obtained by substitution. It is readily soluble in ether, chloroform, and alcohol, sparingly in cold water, but much less so than the fumaroïd acid. It melts at 162°. When heated with hydrochloric acid for three hours at 180°, it is converted into the ordinary acid. The salts are more soluble than those of ordinary hexahydroterephthalic acid, and more difficult to obtain crystalline.

Hexahydroterephthalic acid behaves towards bromine like a saturated fatty acid, yielding substitution products without opening the ring; the free acid is with difficulty attacked, the acid chloride

readily, especially in presence of phosphorus compounds, the bromine taking up the α-position to the carboxyl-group.

1 . 4-*Dibromohexahydroterephthalic acid* is obtained together with 1, 4 dibromo-cis-hexahydroterephthalic acid by treating the hexahydro-acid with phosphorus pentachloride, and heating the product with bromine for three hours at 150°. It forms a granular crystalline precipitate, readily soluble in ether and alcohol, almost insoluble in water. When reduced, it yields both hexahydro-acids and is converted by alcoholic potash into dihydroterephthalic acid. The *methyl salt* crystallises in large, strongly refractive, monoclinic prisms; $a : b : c = 2{\cdot}2742 : 1 : 0{\cdot}89288$; $\beta = 87° 54'$. It melts at 150°.

1 . 4-*Dibromo-cis-hexahydroterephthalic acid* crystallises in extremely slender needles, rather readily soluble in cold water, very readily in alcohol, ether, and acetone. The *methyl salt* crystallises in hair-like needles, melts at 68°, dissolves very readily in alcohol and ether, less in light petroleum. The *anilide* crystallises in long prisms or in hair-like needles, and melts at 200° with decomposition.

1-*Bromohexahydroterephthalic acid* is prepared in a manner exactly similar to the dibromo-acid, except that rather more than half the amount of bromine is used and the mixture is heated for five hours in a water-bath. The product of the reaction, containing unchanged hexahydro-acid, two bromo- and two dibromo-acids, is dissolved in sodium carbonate, filtered and diluted so that the volume amounts to 1 litre (6 grams of acid having been used). It is acidified, kept for 24 hours, when the fumaroïd dibromo-acid separates, filtered, saturated with sodium chloride, and extracted 10 times with ether. The extract is shaken with sodium carbonate, and the aqueous solution, after being freed from dissolved ether, is acidified. The furmaroïd bromo-acid separates first as a granular precipitate, then the cis-dibromo-acid; when no more of the latter separates, the whole is quickly filtered, as the greater part of the cis-monobromo-acid is still in solution. The precipitate is washed with cold water, and the residue, after being freed from hexahydro-acid, is converted into methyl salts. The mixed methyl salts of the fumaroïd and cis-monobromo- and the fumaroïd dibromo-hexahydro-acids are dissolved in light petroleum, and the crystals which separate dissolved in methyl alcohol; the salt of the dibromo-acid separates first, then that of the fumaroïd bromo-acid. This crystallises in monoclinic plates; $a : b : c = 0{\cdot}34724 : 1 : 0{\cdot}46944$; $\beta = 82° 50'$; and melts at 70—71°. The free acid was not obtained pure. When boiled with aqueous soda, it yields the same tetrahydro-acid as does the fumaroïd 2-monobromo-acid.

1-*Bromo-cis-hexahydroterephthalic acid* separates very slowly from its aqueous solution in short, spear-formed crystals melting at about 205°. The *methyl salt* could not be crystallised. The *anilide* forms hair-like needles, rather soluble in hot glacial acetic acid.

When tetrahydroterephthalic acid hydrobromide (2-bromohexahydroterephthalic acid) is converted into the chloride, and treated with bromine, a dibromohexahydro-acid, quite distinct from those obtained by substitution from the hexahydro-acid, is formed. It could not be identified with the dibromide of the tetrahydro-acid, as the methyl salt could not be crystallised, which is probably due to the

presence of geometrical isomerides. When reduced with zinc and glacial acetic acid, it is converted into the tetrahydro-acid. The paper concludes with a reply to Geuther (this vol., p. 579). N. H. M.

Preparation and Oxidation-products of Nitrocymene. By H. G. Söderbaum and O. Widman (*Ber.*, **21**, 2126—2129).—Nitrocymene [Me : NO_2 : Pr = 1 : 2 : 4] is best prepared by dropping a little more than the calculated quantity of nitric acid (sp. gr. 1·4) mixed with $1\frac{1}{2}$ times its weight of concentrated sulphuric acid, into the cymene contained in a well-cooled retort, the whole being shaken during the process. The temperature is kept at 20—25° at first, and afterwards raised to 40°. The mixture is then shaken until it becomes cold, water added, and the nitro-derivative washed with soda, then with water, and distilled with steam. The product contains paratolyl methyl ketone, which can be separated by fractional distillation with steam, the ketone distilling first. The pure nitro-compound is a slightly yellow oil with an aromatic odour; it yields cymidine (carvadrylamine) when reduced with tin and hydrochloric acid. *Cymidine hydrochloride*, $C_{10}H_{15}N,HCl$, crystallises in flat, shining needles. The *sulphate*, $(C_{10}H_{15}N)_2,H_2SO_4 + H_2O$, loses its water at 80°. Orthonitroparahydroxyisopropylbenzoic acid and terephthalic acid are formed when nitrocymene is oxidised with alkaline permanganate.

F. S. K.

Cholesterin. By F. Reinitzer (*Monatsh.*, **9**, 421—441).—The bromo-derivative of acetylcholesterin has the formula $C_{27}H_{45}Br_2OAc$, and, as it is an additive product, the formula of cholesterin is $C_{27}H_{46}O$, but it is probable that several homologous cholesterins occur in the animal organism.

Melted cholesteryl acetate, $C_{27}H_{45}OAc$, shows the colour phenomena before and not after solidifying, as is stated by Raymann (Abstr., 1887, 926). It exists in three modifications: the first is obtained by crystallising it from solvents; when the crystals are warmed, they become clouded before melting, owing to formation of a mass of crystals of the third modification. The second modification is obtained in the form of sphero-crystals when the melted substance is quickly cooled; if xylene is added, the single crystals can be obtained: they are large, thin, monosymmetric leaves, and behave like the first modification when heated. The third modification is formed when either of the other two is heated or when the melted substance is cooled slowly. Cholesteryl acetate is decomposed when heated above its melting point, but a small portion sublimes unchanged. It is not decomposed by water. (Compare Raymann, *loc. cit.*)

Bromocholesteryl acetate is sparingly soluble in alcohol, but readily soluble in ether, and crystallises from alcoholic ether in long, thin, shining plates. It forms monosymmetric crystals melting at 118° (corr.), and asymmetric crystals melting at 115·8° (corr.); it turns yellow when melted, and cannot be again obtained in crystals. It yields cholesterin when reduced with sodium amalgam in ethereal solution, but some other compound appears to be also formed. It gives a yellow, amorphous compound when treated with potash. Cholesteryl benzoate, $C_{27}H_{45}OBz$, melts at 146·6° (corr.) to a turbid

liquid. It exists in three modifications:—The first modification is obtained by crystallisation from solvents; the crystals belong to the tetragonal system, melt at a higher temperature than the other two modifications, and remain clear when heated. The second modification is formed when the melted substance is quickly cooled; it melts at a slightly lower temperature than the third modification, and forms flat needles or small leaves belonging to the rhombic system. The third modification is obtained when the melted substance is cooled slowly; it forms thin, broad leaves. After heating to 178·5°, cholesteryl benzoate shows similar colour phenomena on cooling to those observed in the case of the acetate.

Nitrocholesterin is obtained when a hot, saturated glacial acetic acid solution of cholesterin is treated with nitric acid—sp. gr. 1·54. It is a reddish-yellow, tasteless, odourless, amorphous compound, melts at 93—94° with decomposition, and is readily soluble in alcohol, ether, chloroform, benzene, glacial acetic acid, ammonia, and alkalis, but insoluble in water. F. S. K.

Alkyl-derivatives of Benzylamine. Reduction of Amarine. By H. Zaunschirm (*Annalen*, **245**, 279—288; compare Fischer, this vol., p. 50).—*Benzylidene-ethylamine*, CHPh:NEt, prepared by mixing benzaldehyde with an aqueous 33 per cent. solution of ethylamine, is a clear oil of a penetrating odour, boils at 195° (uncorr.) at 749 mm. pressure, is readily soluble in ether and alcohol, and is decomposed by dilute acids into its constituents. When reduced by sodium amalgam, *ethylbenzylamine*, $CH_2Ph{\cdot}NHEt$, is formed. This boils at 191—194° (uncorr.) under 740 mm. pressure, has an odour like that of benzylamine, dissolves readily in alcohol and ether, and reacts strongly alkaline. The salts dissolve readily in water and alcohol. The *platinochloride*, $(C_9H_{13}N)_2{\cdot}H_2PtCl_6$, crystallises in orange-coloured needles.

Benzylidenemethylamine, CHPh:NMe, resembles the ethylamine compound. *Methylbenzylamine*, $CH_2Ph{\cdot}NHMe$, boils at 184—185° (uncorr.) under 749 mm. pressure; the *platinochloride* was prepared.

Propyl-, isobutyl-, and amyl-benzylamine are strong bases and have an odour of fish; the salts dissolve readily in water and alcohol; the nitroso-derivatives are oils.

Benzylidenepropylamine, CHPh:NHPr, boils at 208—210° (uncorr.) under 744 mm. pressure. *Propylbenzylamine*, $CH_2Ph{\cdot}NHPr$, boils at 210° under 741 mm. pressure; the *platinochloride* crystallises in large, orange-coloured prisms.

Benzylideneisobutylamine, $CHPh{:}N{\cdot}CH_2{\cdot}CHMe_2$, boils at 217—218° (uncorr.) under 735 mm. pressure. *Isobutylbenzylamine*, $C_{11}H_{17}N$, boils at 217—220° (uncorr.) under 741 mm. pressure.

Amylbenzylamine, $CH_2Ph{\cdot}NH{\cdot}C_5H_{11}$, boils at 240° (uncorr.) under 745 mm. pressure; the *platinochloride* crystallises well.

Ethylbenzyldithiocarbamic acid, $CH_2Ph{\cdot}NEt{\cdot}CSSH$, is obtained by treating an alcoholic solution of ethylbenzylamine with carbon bisulphide; after boiling for several hours, the carbon bisulphide and alcohol are distilled off. It melts at 114°, dissolves readily in benzene and carbon bisulphide, sparingly in ether and light petroleum.

When amarine is reduced by sodium amalgam, a compound melting

at 163° is obtained which, when heated with dilute sulphuric acid, is converted into benzaldehyde and a base melting at 108—113°.

Lophine is reduced by sodium amalgam with formation of an isomeric amarine. (Kohler, *Dissertation*, Erlangen, 1888.)

N. H. M.

Ammonia-derivatives of Cumaldehyde. By C. Uebel (*Annalen*, **245**, 289—310).—*Cuminylaniline*, $C_6H_4Pr{\cdot}CH_2{\cdot}NHPh$, is prepared by dissolving cuminaniline (Schiff, *Annalen*, Suppl. **3**, 357) in about 10 parts of alcohol and gradually adding rather more than the calculated amount of 3 per cent. sodium amalgam, the whole being kept cool if necessary; the reaction is completed by heating for a short time on the water-bath in a reflux apparatus. The product is treated with a little water, most of the alcohol distilled off, and the residue steam distilled. The yellow oil is dissolved in ether, dried, freed from ether, and made to crystallise by means of a freezing mixture. It crystallises from alcoholic ether in well-formed, triclinic plates, melts at 41·5°, and dissolves very readily in alcohol and ether. The *hydrochloride* crystallises from dilute alcohol in slender, lustrous needles; the *nitroso-derivative*, $C_6H_4Pr{\cdot}CH_2{\cdot}NPh{\cdot}NO$, crystallises from absolute alcohol in long, yellowish needles melting at 94·5° (uncorr.).

Cumintoluidine, $C_6H_4Pr{\cdot}CH{:}N{\cdot}C_6H_4Me$, is obtained by adding paratoluidine to an alcoholic solution of cumaldehyde; the mixture is warmed a little on a water-bath, treated with alcohol until a clear solution is obtained, and left to crystallise. It forms large, lustrous, bright-yellow, transparent plates, probably triclinic, and melts at 51°. It does not form salts, but is decomposed when heated with alkalis or dilute acids.

Cuminyltoluidine, $C_6H_4Pr{\cdot}CH_2{\cdot}NH{\cdot}C_6H_4Me$, prepared by reducing cumintoluidine with sodium amalgam, crystallises in well-formed, clear, triclinic plates, melts at 36° (uncorr.), and distils above 210° in a vacuum without decomposition. It is very readily soluble in alcohol and ether, insoluble in water. The *hydrochloride* crystallises from hot water in slender, white needles, very readily soluble in alcohol. The *nitroso-derivative* crystallises from alcoholic ether in lustrous, bright-yellow, prismatic needles melting at 67°.

Cuminamidophenol, $C_6H_4Pr{\cdot}CH{:}N{\cdot}C_6H_4{\cdot}OH$, is formed when amidophenol is added to an alcoholic solution of cumaldehyde. It crystallises in well-formed, lustrous, rhombic prisms, of a moss-green colour, melts at 183° with decomposition, and, when dry, remains unchanged.

Cuminylamidophenol, $C_6H_4Pr{\cdot}CH_2{\cdot}NH{\cdot}C_6H_4{\cdot}OH$, separates in greyish-white plates when carbonic anhydride is passed through the solution of the sodium salt; it melts with decomposition at 107—108° (uncorr.), dissolves very readily in alcohol and ether, but cannot be easily recrystallised owing to its instability. The *hydrochloride* forms white plates readily soluble in alcohol and in hot water; the *nitroso-compound* is a yellowish-brown, crystalline substance, readily soluble in alcohol and ether.

Cuminamidodimethylaniline, $C_6H_4Pr{\cdot}CH{:}N{\cdot}C_6H_4{\cdot}NMe_2$, obtained by the action of cumaldehyde on freshly prepared amidodimethylaniline (Wurster, Abstr., 1879, 626), crystallises in intensely yellow, slender

needles, melts at 100·5° (uncorr.), is readily soluble in alcohol and ether, and insoluble in water.

Cuminylamidodimethylaniline, $C_6H_4Pr{\cdot}CH_2{\cdot}NH{\cdot}C_6H_4{\cdot}NMe_2$, crystallises in large, well-formed, prismatic plates, melts at 39° (uncorr.), and dissolves readily in alcoholic ether. The *hydrochloride* separates from the alcoholic solution in long, slender needles of a silky lustre. The *nitroso-derivative* crystallises in long, gold-coloured needles, melts at 87°, and dissolves readily in alcohol and ether.

Hydrocuminamide, $C_6H_7{\cdot}C_6H_4{\cdot}CH(N{:}CH{\cdot}C_6H_4Pr)_2$ (Borodin, this Jour., 1874, 273, and Sieveking, *Annalen*, **106**, 259), is prepared by passing dry ammonia through a solution of cumaldehyde in an equal volume of absolute alcohol and much absolute ether for several hours under pressure. The vessel containing the mixture is well closed, and kept for some time in a freezing mixture, when hydrocuminamide separates in groups of needles. The ether is poured off, and the solution as well as the undissolved portion put into a desiccator which is pumped out several times and kept in a cold place. It forms stellate groups of needles, melts at 65°, and is very readily soluble in alcohol, less so in ether. When pure, it remains unchanged if kept in closed vessels, but becomes yellow and resinous when exposed to moist air. When reduced with sodium amalgam, it is converted into isopropylbenzylamine and diisopropylbenzylamine, which are separated from each other by distillation with steam.

Isopropylbenzylamine, which is found in the distillate, is identical with Rossi's cumylamine (compare Goldschmidt and Gessner, Abstr., 1887, 1039). It boils at 226—228° (uncorr.). When exposed to the air, it absorbs carbonic anhydride very readily, yielding a *carbonate* which crystallises in large, transparent plates. The *platinochloride* crystallises from alcohol, in which it is readily soluble, in gold-coloured plates.

Diisopropylbenzylamine, $NH(CH_2{\cdot}C_6H_4Pr)_2$, forms white, compact crystals, melts at 168°, boils without decomposition at 280—300° under 100 mm. pressure. It is readily soluble in alcohol, insoluble in water. The *hydrochloride* crystallises in large, lustrous plates, very readily soluble in alcohol; the *platinochloride* forms slender, yellow needles. The *nitrosamine*, $NO{\cdot}N(CH_2{\cdot}C_6H_4Pr)_2$, crystallises in long, pale-yellow needles, readily soluble in alcohol, sparingly in ether.

N. H. M.

Nitramines derived from Alkyl Aromatic Diamines. By P. VAN ROMBURGH (*Rec. Trav. Chim.*, **6**, 251—254).—When methylmetaphenylenediamine is boiled with fuming nitric acid, *trinitrophenylenedimethyldinitramine*, $C_6H(NMe{\cdot}NO_2)_2(NO_2)_3$, is formed.

Tetranitrodimethyldinitramidobenzophenone,

$$CO[C_6H_2(NO_2)_2NMe{\cdot}NO_2]_2,$$

is obtained by boiling tetramethyldiamidobenzophenone with nitric acid (sp. gr. = 1·40 or 1·42); it forms pale-yellow crystals which decompose at 210°. Boiling phenol acts on it with formation of *tetranitrodimethyldiamidobenzophenone*, $CO[C_6H_2(NO_2)_2{\cdot}NHMe]_2$. This is yellow, and melts at 225° with decomposition.

When dinitrodimethylaniline is treated with nitric acid (sp. gr. = 1·36) in the cold, two isomeric trinitrodimethylanilines, melting at 196° and 154° respectively, are formed. (Compare *Rec. Trav. Chim.*, **2**, 105.) Boiling fuming nitric acid converts the above di- and tri-nitro-derivatives into a yellow crystalline substance, probably $OH\cdot C_6H(NO_2)_3\cdot NMe\cdot NO_2$. N. H. M.

Thio-derivatives of some Secondary and Tertiary Amines. By E. Holzmann (*Ber.*, **21**, 2056—2071).—*Oxydimethylaniline*, $O(C_6H_4\cdot NMe_2)_2$, is formed when an alcoholic solution of 1 mol. of thiodimethylaniline in presence of ammonia is boiled with an ammoniacal alcoholic solution of silver nitrate for about one hour. It crystallises in stellate groups of needles melting at 119°, and is soluble in alcohol, ether, and benzene, readily soluble in acids. The *platinochloride*, $O(C_6H_4\cdot NMe_2)_2,H_2PtCl_6$, is a bright yellow, microcrystalline substance, sparingly soluble in hot alcohol and water. The *picrate* is a yellow, crystalline precipitate, melts at 150°, dissolves readily in hot alcohol and benzene; when heated it detonates.

Thiodiethylaniline, $S(C_6H_4\cdot NEt_2)_2$, is prepared by dissolving diethylaniline (4 mols.) in light petroleum, and gradually adding to the well-cooled solution a solution of sulphur dichloride (1 mol.) in light petroleum. It crystallises from alcohol in yellowish needles, melts at 79·5—80°, is insoluble in water, readily soluble in warm alcohol, ether, benzene, and glacial acetic acid. The *platinochloride* is a yellow, flaky precipitate, insoluble in water, very sparingly soluble in alcohol. The *picrate* forms sulphur-coloured, slender, lustrous needles, melts at 175°, and is sparingly soluble.

Oxydiethylaniline, $O(C_6H_4\cdot NEt_2)_2$, crystallises in colourless needles, melts at 89°, is insoluble in water, readily soluble in warm alcohol, ether, and benzene. When pure, it is stable when exposed to air. The *platinochloride* is a yellow, flaky, sparingly soluble substance. The *picrate* forms slender, yellow crystals, melts at 174°, and is sparingly soluble.

Dithiodiphenylamine, $S_2<^{C_6H_4}_{C_6H_4}>NH$, is formed when a solution of 10 grams of diphenylamine in an excess of light petroleum is well-cooled and treated with a solution of 2·5 grams of sulphur chloride in light petroleum. The greenish product is dissolved in benzene and boiled with benzene in a reflux apparatus. It crystallises in slender, yellow needles melting at 59—60°. It dissolves readily in warm alcohol, ether, and benzene, and is insoluble in water.

Thiodiphenylamine (Bernthsen, Abstr., 1884, 595) is obtained by treating a well-cooled solution of diphenylamine in benzene with a similar solution of sulphur dichloride. It crystallises in almost colourless plates, melts at 180°, and boils at 370°.

Methylthiodiphenylamine, $S<^{C_6H_4}_{C_6H_4}>NMe$, prepared by the action of sulphur dichloride on methyldiphenylamine, forms slender, bright yellow needles, melts at 78—79°, and decomposes when heated above 360°. It is insoluble in water, rather soluble in warm benzene, sparingly soluble in boiling alcohol and ether. The compound is an

isomeride of that prepared by Bernthsen (*loc. cit.*) from thiodiphenylamine, methyl iodide, and methyl alcohol (compare Mohlau and Krohn, this vol., p. 364).

When thiodimethylaniline is heated with 10 parts of freshly reduced copper at 300°, dimethylaniline is formed. Thiodiphenylamine, when heated with copper, yields carbazole (Goske, Abstr., 1887, 372), whilst dithiodiphenylamine yields diphenylamine. Methylthiodiphenylamine (from thiodiphenylamine) is converted by copper into carbazole; methylthiodiphenylamine from methyldiphenylamine yields no carbazole but methyldiphenylamine.

N. H. M.

Action of Aniline on Quinonephenylimide and Diphenylparazophenylene. Synthesis of Dianilidoquinoneanil and Azophenine. By E. v. Bandrowski (*Monatsh.*, **9**, 414—420; compare this vol., p. 943).—Quinonephenylimide and aniline react very readily, producing dianilidoquinoneanil and parahydroxydiphenylamine; the yield of the former is almost theoretical.

Diphenylparazophenylene and aniline yield azophenine and diphenylparaphenylenediamine. The quantity of azophenine produced is 59·9 per cent. of the diphenylparazophenylene employed.

F. S. K.

Action of Paratoluidine and of Aniline on Phloroglucinol. By G. Minunni (*Ber.*, **21**, 1984—1991).—*Triparatolyltriamidobenzene*, $C_6H_3(NH\cdot C_7H_7)_3$, is formed when a mixture of paratoluidine and phloroglucinol is heated at 200—210°. It crystallises in slender, white needles melting at 186—187°, is very sparingly soluble in cold alcohol, sparingly soluble in hot alcohol, but relatively easily soluble in warm benzene or ether. It is soluble in acids; a cold concentrated sulphuric acid solution turns bluish-green and finally black when heated, and with sodium nitrite gives a bluish-green coloration which changes to brown on adding a considerable quantity of the reagent. The base forms two hydrochlorides. The *monacid* salt, $C_{27}H_{27}N_3,HCl$, is obtained in the form of a yellowish, seemingly amorphous precipitate when dry hydrogen chloride is passed into a benzene solution of the base. The *diacid* salt, $C_{27}H_{27}N_3,2HCl$, crystallises in small needles when a concentrated hydrochloric acid solution is evaporated slowly. Both salts are decomposed by an excess of water. The *platinochloride*, $(C_{27}H_{27}N_3)_2,H_2PtCl_6$, is a yellow, crystalline compound which is insoluble in ordinary solvents and is decomposed by boiling water. The triacetyl-derivative, $C_6H_3(NAc\cdot C_7H_7)_3$, crystallises from warm alcohol in microscopic leaves, melts at 192—193°, is readily soluble in benzene, sparingly soluble in ether, and insoluble in water. The *tribenzoyl*-derivative, $C_6H_3(NBz\cdot C_7H_7)_3$, crystallises from boiling alcohol in microscopic prisms melting at 281—282°. It is almost insoluble in cold ether, only very sparingly soluble in warm ether, and sparingly in alcohol and benzene. It is soluble in concentrated sulphuric acid and is precipitated unchanged by water.

Triphenyltriamidobenzene, $C_6H_3(NHPh)_3$, prepared by heating phloroglucinol with aniline at 210°, crystallises from alcohol in slender, white needles, melting at 193°. It is sparingly soluble in cold, but readily soluble in hot alcohol or benzene, and still more readily

soluble in ether. Ammonia precipitates it unchanged from the hydrochloric acid solution. A concentrated sulphuric acid solution becomes violet-red and finally black when heated; in a sulphuric acid solution, sodium nitrite produces a violet-red coloration which changes to yellowish-brown on adding a considerable quantity of the reagent. The *hydrochloride*, $C_6H_3(NHPh)_3,HCl$, is a yellow powder, melts below 100°, and is decomposed by warm water. The *platinochloride*, $(C_{24}H_{21}N_3)_2,H_2PtCl_6$, is yellow, crystalline, insoluble in ether and benzene, and sparingly soluble in boiling alcohol, and melts at about 250° with decomposition. The *triacetyl*-derivative, $C_6H_3(NPhAc)_3$, crystallises from boiling alcohol in microscopic needles, melts at 172—173°, is sparingly soluble in cold, but very readily soluble in hot alcohol or ether. The *tribenzoyl*-derivative, $C_6H_3(NPhBz)_3$, crystallises from alcoholic benzene in white, microscopic needles melting above 300°. It is soluble in hot, but only sparingly soluble in cold alcohol or benzene. F. S. K.

Disazo-compounds. By R. Nietzki and J. Diesterweg (*Ber.*, **21**, 2143—2147).—Diazoazobenzene combines with aniline to form a yellowish-red diazoamido-compound melting at 119°. When this substance is dissolved in aniline, mixed with aniline hydrochloride, and kept for about 24 hours at 40—50°, the greater part is converted into ordinary amidoazobenzene, but a small quantity of amidodisazobenzene is also formed. The latter is isolated by precipitating with hydrochloric acid, dissolving the precipitate in alcoholic ammonia, and reprecipitating with hydrochloric acid. The mixture of hydrochlorides is decomposed with alkali and the bases converted into the acetyl-derivatives. The acetylamidodisazobenzene, which is more sparingly soluble, is separated from the acetylamidoazobenzene by partial recrystallisation from alcohol. The yield is about 1 per cent.

Acetylamidodisazobenzene, $PhN_2{\cdot}C_6H_4{\cdot}N_2{\cdot}C_6H_4{\cdot}NHAc$, crystallises from hot alcohol or glacial acetic acid in orange-yellow plates, melts at 227°, and gives a dark green coloration with concentrated sulphuric acid.

Amidodisazobenzene is obtained by boiling the acetyl-derivative with alcoholic potash. It crystallises in yellow plates, melts at 170°, is very sparingly soluble in alcohol, and dissolves in concentrated sulphuric acid, forming a dark red solution which turns a deep blue when a few drops of water are added.

Disazobenzene, $PhN_2{\cdot}C_6H_4{\cdot}N_2Ph$, is formed when the amido-derivative is treated with potassium nitrite and sulphuric acid in alcoholic solution and the mixture boiled for some time. It crystallises from ether or benzene in yellowish-red, well-defined needles, melts at 98°, and dissolves in concentrated sulphuric acid, forming a yellow solution.

α-Amidonaphthalenedisazobenzene, $PhN_2{\cdot}C_6H_4{\cdot}N_2{\cdot}C_{10}H_6{\cdot}NH_2$, prepared by mixing solutions of diazoazobenzene hydrochloride and α-naphthylamine hydrochloride, crystallises from glacial acetic acid in green plates which melt at 170°, and give a deep blue colour with concentrated sulphuric acid. The *acetyl*-derivative,

$$PhN_2{\cdot}C_6H_4{\cdot}N_2{\cdot}C_{10}H_6{\cdot}NHAc,$$

crystallises in yellowish-red plates, melts at 275°, and dissolves in concentrated sulphuric acid, forming a green solution.

Naphthalenedisazobenzene, $PhN_2{\cdot}C_6H_4{\cdot}N_2{\cdot}C_{10}H_7$, prepared by treating amidonaphthalenedisazobenzene with sodium nitrite and sulphuric acid in alcoholic solution and boiling the mixture, crystallises in yellowish-brown plates, melts at 143°, and gives a green solution with concentrated sulphuric acid.

β-Amidonaphthalenedisazobenzene, prepared similarly to the α-compound, crystallises from alcohol in red needles, melts at 164°, and gives a blue solution with sulphuric acid. The *acetyl*-derivative crystallises in red plates, melts at 206°, and dissolves in concentrated sulphuric acid with a red colour. F. S. K.

Carbodiimides of the Aromatic Series and Phenylhydrazine. By R. Wessel (*Ber.*, **21**, 2272—2278).—*Carbodiphenylaminehydrazide*, $N_2HPh{:}C(NHPh)_2$, is obtained when carbodiphenylamide and phenylhydrazine in molecular proportion are heated together at 120° for half an hour. It crystallises from boiling alcohol in slightly reddish needles, melts at 204°, and turns red on exposure to the air. It is readily soluble in boiling alcohol, benzene, chloroform, &c., moderately soluble in ether, but almost insoluble in light petroleum. The salts are colourless and crystalline. The *hydrochloride*, $C_{19}H_{18}N_4,HCl$, crystallises from warm alcohol in needles, and is insoluble in ether and light petroleum, but readily soluble in water, dilute hydrochloric acid, and boiling alcohol. The *platinochloride*, $(C_{19}H_{18}N_4)_2,H_2PtCl_6$, crystallises from warm alcohol in yellow, lustrous needles, is almost insoluble in water, and decomposes when heated. The *sulphate*, $C_{19}H_{18}N_4,H_2SO_4$, separates from alcoholic ether in white, crystalline crusts, and is soluble in boiling alcohol and water but almost insoluble in ether.

Carbodiparatolylaminehydrazide, $N_2HPh{:}C(NH{\cdot}C_7H_7)_2$, prepared by heating a mixture of carbodiparatolylimide with phenylhydrazine in molecular proportion at 160—170°, crystallises from warm alcohol in pale-red needles, melts at 138°, and behaves towards solvents like the preceding compound. The *platinochloride*, $(C_{21}H_{22}N_4)_2,H_2PtCl_6$, is very similar to the corresponding salt described above.

The compound, $2C_{13}H_{10}N_2,C_6H_8N_2$, is formed when carbodiphenylaminehydrazide (1 mol.) is heated at 185° for half an hour with phenylhydrazine (1 mol.). It separates from boiling alcohol in white crystals, melts at 200°, is only moderately soluble in boiling alcohol or ether, more readily in boiling benzene, and insoluble in light petroleum. The *hydrochloride*, $(C_{32}H_{28}N_6)_3,4HCl$, crystallises in white plates, and is readily soluble in hot water, dilute hydrochloric acid, and boiling alcohol. The *platinochloride*, $(C_{32}H_{28}N_6)_3,2H_2PtCl_6$, is decomposed when gently heated.

The compound, $2C_{15}H_{14}N_2,C_6H_8N_2$, is obtained by heating 1 mol. of carboditolylaminehydrazide with 1 mol. of phenylhydrazine at 190—195°. It crystallises from alcohol in plates, melts at 163°, and is only very sparingly soluble in boiling ether. The *hydrochloride*, $(C_{36}H_{36}N_6)_3,4HCl$, separates from a hot hydrochloric acid solution of

the base in white crusts. The *platinochloride*, $(C_{26}H_{36}N_6)_3,2H_2PtCl_6$, is decomposed when heated gently.

The compound $C_{13}H_{20}N_2,C_{15}H_{14}N_2,C_6H_6N_2$ is obtained when carbodiphenylaminehydrazide is heated with carbodiparatolylimide. It crystallises from boiling alcohol in white needles, melts at 128°, and is sparingly soluble in warm alcohol or ether. The *hydrochloride*, $(C_{34}H_{32}N_6)_3,4HCl$, crystallises in colourless plates, and is readily soluble in hot water and boiling alcohol. The *platinochloride*,

$$(C_{34}H_{32}N_6)_3,2H_2PtCl_6,$$

decomposes when heated gently.

The compound $C_6H_8N_2,C_{13}H_{10}N_2,C_7H_5NS$, prepared by heating carbodiphenylaminehydrazide with phenylthiocarbimide at 190°, crystallises from boiling alcohol in white needles, melts at 175°, and is moderately soluble in boiling alcohol or ether. F. S. K.

Action of Carbamide on Hydrazines. By A. Pinner (*Ber.*, **21**, 2329—2331).—The compound obtained by heating phenylsemicarbazide (Pinner, this vol., p. 687) is *diphenylurazine*, $C_{14}H_{12}N_4O_2$; when boiled with acetic anhydride for a few seconds, it yields an *acetyl*-derivative, $C_{14}H_{11}N_4O_2Ac$, which separates from benzene in small, nodular crystals melting at 173°. The *diacetyl*-derivative, $C_{14}H_{10}N_4O_2Ac_2$, obtained by heating diphenylurazine with acetic anhydride and sodium acetate, crystallises in stellate needles, melts at 153°, and is readily soluble in alcohol and benzene, but insoluble in water and light petroleum.

Ethyldiphenylurazine, $C_{14}H_{11}N_4O_2Et$, prepared by heating an alcoholic solution of the urazine with potash (2 mols.) and ethyl iodide (2 mols.), crystallises in large, shining prisms, melts at 137°, and is readily soluble in alcohol, but insoluble in ether.

The constitution of diphenylurazine is probably $<\begin{matrix}NPh\cdot NH\cdot CO\\NPh\cdot CO\cdot NH\end{matrix}>$.

F. S. K.

Xanthogallol. By C. A. Theurer (*Annalen*, **245**, 327—356).—Xanthogallol is very stable towards oxidising agents, and can be crystallised from strong nitric acid; it is very readily reduced. When heated at 130° it sublimes with partial decomposition.

When 5 grams of xanthogallol dissolved in 25 grams of glacial acetic acid is cooled and treated with 5 grams of aniline diluted with glacial acetic acid, the compound $C_{18}H_4Br_{11}O_6(NHPh)_4$ is formed. This crystallises in large, greenish-yellow prisms and melts at 204—206°.

The *paratoluidine-derivative*, $C_{18}H_4Br_{11}O_6(NH\cdot C_7H_7)_4$, is more readily soluble in alcohol than the aniline compound.

The *chloromethoxy-derivative*, $C_{18}H_4Br_{11}Cl_3O_3(OMe)_3$, is formed when hydrogen chloride is passed through a cooled solution of 4 grams of xanthogallol in 80 grams of methyl alcohol. It crystallises in large, well-formed, colourless crystals, melts at 86°, dissolves readily in methyl alcohol, alcohol, and glacial acetic acid, and is almost insoluble in water. It does not react with aniline, and is decomposed by dilute alkalis.

The *chlorethoxy-derivative*, $C_{18}H_4Br_{11}Cl_3O_3(OEt)_3$, crystallises in large prisms of a vitreous lustre, melting at 75°; it is more difficult to saponify than the methoxy-derivative.

The *bromomethoxy-derivative*, $C_{15}H_4Br_{14}O_3(OMe)_3$, prepared in a manner similar to the chloromethoxy-compound, using hydrogen bromide, forms colourless prisms melting at 113°. When boiled with dilute soda, filtered, and treated with excess of strong hydrochloric acid, the compound $C_6H_2Br_4O_2(OMe)_2$ is obtained. The latter melts at 105°, and is very readily soluble in alcohol, ether, glacial acetic acid and benzene, less soluble in water. When dissolved in methyl alcohol, and the solution saturated with hydrogen chloride, the compound $C_6HBr_4Cl(OMe)_4$ is formed. This forms large, hard prisms of vitreous lustre, melting at 77°.

The *aniline-derivative of xanthogallol*, $C_6H_3Br_4O_4Me_2{\cdot}NH_2Ph$, is obtained by treating the acid $C_6H_2Br_4O_2(OMe)_2$ with aniline; it forms white crystals.

In the preparation of the ethoxy-derivative from xanthogallol, the compound $C_{18}H_4Br_{11}Cl_3O_6$ is obtained. It forms large, yellow crystals, melts at 104°, and dissolves readily in alcohol and benzene.

Trihydroxyxanthogallol crystallises with 7 mols. H_2O in long, matted needles melting at 72°; the anhydrous crystals melt at 131°. The *aniline-derivative*, $C_{18}H_7Br_{11}O_9(NH_2Ph)_8$, crystallises in small, white needles, very readily soluble in alcohol. The *toluidine-derivative*, $C_{18}H_7Br_{11}O_9(NH_2{\cdot}C_7H_7)_8$, forms small, white plates.

When xanthogallol is suspended in water and treated with soda, it gradually dissolves, whilst the liquid becomes milky and gradually deposits a pulverulent precipitate mixed with oily drops. It is extracted with ether, and the oil so obtained dissolved in alcohol. On evaporating the alcohol, crystals of the *hexabromodihydrobenzene*, $C_6H_2Br_6$, separate. This forms long, white prisms, melts at 139°, and is sparingly soluble in alcohol and light petroleum, more soluble in benzene; it is very stable, and is not attacked by alkalis or by sulphuric and nitric acids. Alcoholic potash acts on it, eliminating hydrogen bromide. The alkaline solution, from which the neutral substance was extracted, contains the sodium salt of an acid. The *free acid* forms slender, white needles, and melts at 124°; the *barium salt*, $C_6H_2Br_2BaO_3 + 3H_2O$, was prepared.

Xanthogallol may be considered as derived from 3 mols. of tribromopyrogallol by the elimination of 3 mols. H_2O and addition of 2 mols. of bromine and 1 mol. of hydrogen bromide. It would then have the formula $C_6HOBr_5<\begin{matrix}O{\cdot}C_6H_2OBr_4\\O{\cdot}C_6HOBr_5\end{matrix}>O$. This formula explains the various reactions of xanthogallol, and the formation of derivatives containing six carbon-atoms. The constitution of these compounds and of trihydroxyxanthogallol is also discussed.

N. H. M.

Acetopropylbenzene, Acetocumene, and their Derivatives. By O. WIDMAN (*Ber.*, **21**, 2224—2233).—*Paracetopropylbenzene*, $C_6H_4Pr^a{\cdot}COMe$, prepared by treating propylbenzene with acetic chloride in presence of aluminium chloride, is a colourless, mobile

liquid with a strong aromatic odour. It boils at 259° (765 mm.), and distils with slight decomposition.

Paracetocumene, $C_6H_4Pr^{\beta}\cdot COMe$, prepared in like manner, boils at 252—254° (756 mm).

Acetopropylbenzeneoxime, $C_6H_4Pr^{\alpha}\cdot CMe{:}NOH$, obtained by warming an alcoholic solution of acetopropylbenzene with hydroxylamine, crystallises from light petroleum in large, well-defined, colourless, very readily soluble plates melting at 43—44° (sometimes at about 52—54°). The *hydrazide* crystallises from warm, light petroleum, in which it is readily soluble, in yellowish, hexagonal, axe-shaped plates melting at 92° with decomposition.

Acetocumeneoxime, $C_6H_4Pr^{\beta}\cdot CMe{:}NOH$, is obtained, together with a small quantity of an oily, isomeric compound, by warming an alcoholic solution of acetocumene with hydroxylamine. It crystallises from light petroleum in rhombic plates melting at 70—71°. The *hydrazide* crystallises from light petroleum in long, colourless or yellowish, hexagonal plates melting at 81—82°.

Nitracetopropylbenzene, $[Pr^{\alpha} : NO_2 = 1 : 2]$, prepared by treating acetopropylbenzene with a mixture of concentrated sulphuric acid and fuming nitric acid, is a yellowish oil. It yields nitropropylbenzoic acid when oxidised with alkaline potassium permanganate.

Nitracetocumene, $[Pr^{\beta} : NO_2 = 1 : 2]$, crystallises in quadratic prisms or domes, melts at 49°, and is very readily soluble in benzene, but only sparingly in light petroleum. It gives metanitrocuminic acid and nitrohydroxyisopropylbenzoic acid when oxidised with alkaline permanganate.

Nitracetopropylbenzeneoxime, $NO_2\cdot C_6H_3Pr^{\alpha}\cdot CMe{:}NOH$, crystallises in needles or prisms, melts at 86°, and is readily soluble in benzene and sparingly soluble in light petroleum. It yields nitropropylbenzoic acid when oxidised with alkaline potassium permanganate.

Nitracetocumeneoxime, $NO_2\cdot C_6H_3Pr^{\beta}\cdot CMe{:}NOH$, is a colourless, crystalline compound, melts at 116—117°, and is readily soluble in warm benzene, but only sparingly in light petroleum.

Amidoacetopropylbenzene, prepared by reducing a dilute soda solution of nitracetopropylbenzeneoxime with ferrous sulphate, crystallises in yellow prisms, melts at 116—117°, and is readily soluble in alcohol and benzene, but almost insoluble in boiling light petroleum.

Amidoacetocumeneoxime, prepared in like manner, crystallises from light petroleum in prisms melting at 95°.

Nitracetopropylbenzenehydrazide, $NO_2\cdot C_6H_3Pr^{\alpha}\cdot CMe{:}N_2HPh$, crystallises from alcohol in slender, red needles, melts at 138—139°, and is sparingly soluble in boiling alcohol.

Nitracetocumenehydrazide melts at 138°.

Acetopropylbenzene is only very slowly oxidised by warm potassium permanganate, but in alkaline solution oxidation takes place extremely rapidly. Terephthalic acid is formed in both cases.

Propylbenzoic acid is best prepared by boiling 3 grams of acetopropylbenzene with 100 c.c. of nitric acid, sp. gr. 1·07, for about 10 hours. The product is distilled with steam, the distillate neutralised with potash and evaporated; the acid is then precipitated with

hydrochloric acid and crystallised from a mixture of benzene and light petroleum. F. S. K.

Benzene-derivatives of High Molecular Weight. By F. KRAFFT (*Ber.*, **21**, 2265—2271).—The best source for pure palmitic acid is Japanese wax; 3 parts of the wax are saponified with a solution of 1 part of potash in 1 part of water, the acid precipitated with hydrochloric acid, and rectified once or twice in a partial vacuum. The silver salts of the higher fatty acids can be readily obtained in a pure and crystalline condition by mixing an alcoholic ammoniacal solution of the acid with a similar solution of silver nitrate; on adding water the salt separates in small plates. Pentadecyl phenyl ketone (compare F. Krafft, Abstr., 1887, 252) emits light when cut or broken.

Pentadecyl paratolyl ketone, $C_{15}H_{31}\cdot CO\cdot C_6H_4Me$, prepared by gradually adding aluminium chloride to a cooled mixture of 1 part of palmitic chloride and 2 parts of toluene, and heating the mixture very slowly until it begins to boil, crystallises from alcohol in needles, melts at 60°, and boils at 262° (15 mm. pressure). It yields paratoluic acid when boiled with nitric acid, sp. gr. 1·12, for several hours, and when broken or rubbed emits bluish-green sparks of great intensity.

Heptadecyl paratolyl ketone, $C_{17}H_{35}\cdot CO\cdot C_6H_4Me$, prepared in like manner from stearic chloride and toluene, melts at 67°, boils at 278° (15 mm. pressure), and is sparingly soluble in cold alcohol, but more readily in ether and benzene. It yields paratoluic acid when oxidised with nitric acid, and emits light when cut or broken.

Pentadecyl xylyl ketone, $C_{15}H_{31}\cdot CO\cdot C_6H_3Me_2$, prepared in like manner from metaxylene and palmitic chloride, melts at 37°, boils at 268—269° (15 mm. pressure), and yields xylylic acid, melting at 126°, and fatty acid when boiled with nitric acid.

Pentadecyl paranisyl ketone, $C_{15}H_{31}\cdot CO\cdot C_6H_4\cdot OMe$, obtained from palmitic chloride and anisoïl, melts at 70·5°, boils at 279—280° (15 mm. pressure), and yields anisic acid when oxidised with nitric acid. It is converted into the corresponding phenol when melted with potash and a little water.

Pentadecyl paraphenetyl ketone, $C_{15}H_{31}\cdot CO\cdot C_6H_4\cdot OEt$, prepared from palmitic chloride and phenetoïl, melts at 69°, boils at 288—289° (15 mm. pressure), and crystallises from cold alcohol, in which it is sparingly soluble, in shining plates. It yields paraethoxybenzoic acid when oxidised with dilute nitric acid.

Pentadecyl dimethylresorcyl ketone, $C_{15}H_{31}\cdot CO\cdot C_6H_3(OMe)_2$, prepared by gradually adding aluminium chloride to a mixture of dimethylresorcinol and palmitic chloride, and heating the mixture extremely slowly from 40° to 100°, crystallises from hot alcohol in plates, melts at 63·5°, and boils at 289—290° (15 mm. pressure). F. S. K.

New Aromatic Diketone. By H. v. PECHMANN and H. MÜLLER (*Ber.*, **21**, 2119—2120).—*Methyl phenyl diketone*, COMe·COPh, prepared from α-nitrosopropiophenone by the action of dilute acids, forms a yellow oil heavier than water. It boils at 214°, distils with steam, and the vapour has a very penetrating odour. *α-Nitroso-*

propiophene is obtained by V. Meyer's method from ethyl methylbenzoylacetate. It crystallises in white needles melting at 113°.

N. H. M.

Action of Amyl Nitrite on Nitrosoketones. By O. Manasse (*Ber.*, **21**, 2176—2177).—*Benzoylacetyl*, COPh·COMe, is formed when nitrosoethyl phenyl ketone is heated with about 1½ mols. of amyl nitrite. It is a yellowish oil. F. S. K.

Nitrotoluic Acids. By S. Niementowski and B. Rozanski (*Ber.*, **21**, 1992—1999).—*Orthonitroparatoluic acid*, [COOH : NO_2 : Me = 1 : 2 : 4], is best prepared by heating orthonitroparatolunitrile with concentrated hydrochloric acid at about 195°. The yield is 82 per cent. It crystallises from boiling water in long, tolerably thick, yellowish needles, melts at 161°, and is almost insoluble in cold, and sparingly soluble in hot water; it is soluble in ether, benzene, and chloroform, and very readily soluble in alcohol. When heated above its melting point, it sublimes, without decomposition, in snow-white, very slender, concentrically grouped needles. The *ammonium* salt crystallises in needles or plates, which are very readily soluble in water. Lead acetate, ferric chloride, and mercuric nitrate give precipitates with a solution of the ammonium salt. The *silver* salt,

$$NO_2·C_6H_3Me·COOAg,$$

crystallises in concentrically grouped needles which are sparingly soluble in boiling water and insoluble in alcohol and cold water. The *barium* salt, $(C_8H_6NO_4)_2Ba + 4H_2O$, crystallises in nacreous leaves, and loses the whole of its water at 135°; it is very sparingly soluble in water and rather sparingly soluble in alcohol. The *calcium* salt, $(C_8H_6NO_4)_2Ca$, crystallises in well-defined yellowish prisms, and is more sparingly soluble in water than the barium salt. The *copper* salt with 1 mol. H_2O crystallises in small, bluish-green scales, is almost insoluble in cold water and alcohol, and sparingly soluble in boiling water. All the salts of orthonitroparatoluic acid explode when heated on platinum foil.

Orthonitroparatoluylamide, obtained by heating the nitrile with an excess of potash, crystallises from boiling benzene or light petroleum in long, slender needles, melts at 153°, is readily soluble in alcohol, and soluble in water, methyl alcohol, and boiling ether. It yields orthonitroparatoluic acid when heated with concentrated hydrochloric acid. Metahomoanthranilic acid (Niementowski, this vol., p. 837) is produced when orthonitroparatoluic acid is reduced with tin and hydrochloric acid. The yield is 46 per cent. F. S. K.

Derivatives of Ethyl Benzoylacetate. By R. Stierlin (*Ber.*, **21**, 2120—2125).—When ethyl benzoylacetate (1 mol.) is treated with dilute potash (1 mol.) and the resulting potassium compound dissolved in much water, cooled, and an aqueous solution of diazobenzene chloride added, a brown oil separates, which is extracted with ether. The oil solidifies when kept to a half-solid mass. It crystallises from alcohol in honey-coloured, lustrous prisms, melts at 65°, is very soluble in alcohol, ether, and benzene, but not in water. The new compound

has the formula $C_{17}H_{16}O_3N_2$, and is probably a hydrazone of the constitution NHPh·N:CBz·COOEt. The *free acid*, $C_{15}H_{12}N_2O_3$, obtained by heating the ethyl salt in a water-bath with dilute potash from three to four minutes, filtering, and acidifying the well-cooled filtrate with sulphuric acid, crystallises from dilute alcohol in slender, yellow needles of a satiny lustre. It melts at 142°, dissolves very readily in alcohol, ether, and benzene, but not in water. The *salts* are bright yellow. When the ethyl salt is heated for a long time with dilute potash, the compound COPh·CH:N·NHPh is formed. This crystallises in lustrous, yellow plates, melts at 129°, and dissolves very readily in alcohol, ether, and benzene, sparingly in hot water. The crystals have a peculiar odour.

The next homologue of the above acid is prepared from paradiazotoluene chloride and ethyl benzoylacetate. It crystallises in slender, yellow needles of a silky lustre, and melts at 169—170°. The *ketone*, COPh·CH:N·NH·C_6H_4Me, crystallises in yellowish-brown plates, melts at 122—123°, and dissolves readily in ether and benzene.

From ethyl benzoylacetate and paranitrodiazobenzene chloride the *ethyl salt*, NO_2·C_6H_4·NH·N:CBz·COOEt, was prepared. It forms very lustrous, yellow plates, melts at 114°, and dissolves readily in ether, alcohol, and benzene.

When the condensation-product from ethyl benzoylacetate and diazobenzene chloride is treated with phenylhydrazine acetate, a *pyrazole-derivative*, $<^{NPh\cdot CO}_{N:CPh-}>C:N\cdot NHEt$, is obtained. This forms red crystals melting at 169°, readily soluble in alcohol and benzene. Warm aqueous soda dissolves it slowly with decomposition.

Ethyl benzoylacetate seems to react with phenylhydrazinesulphonic acid and sodium acetate in a similar manner. N. H. M.

Condensation of Ethyl Benzoylacetate with Succinic Acid. By R. Fittig and A. Schloesser (*Ber.*, **21**, 2133—2137).—A compound, $COOEt\cdot C<^{CPh-}_{CO\cdot CH_2}>CH\cdot COOH$, is formed when ethyl benzoylacetate and sodium succinate react in presence of acetic anhydride. It crystallises in short needles, melts at 112·5°, and is sparingly soluble in water.

Phenythronic acid, $C_{10}H_{10}O_5$, is obtained when the preceding compound is treated with baryta-water. It crystallises from hot water, in which it is rather easily soluble, in long, silky needles, melts at 192°, and yields a diethyl salt which is identical with that obtained from the preceding compound, and can be converted into the preceding compound by treating it carefully with alcoholic potash. When heated above its melting point, phenythronic acid yields carbonic anhydride, phenylmethylfurfuran, and a monobasic acid, *phenuvinic* acid, $C_{12}H_{10}O_3$, which crystallises in long needles, melts at 144—145°, and is insoluble in cold, and only sparingly soluble in hot water. F. S. K.

Constitution of the Compound obtained by Acting on Trimethylpyrogallol with Nitric Acid. By W. Will (*Ber.*, **21**,

2020—2026).—*Methyl gallate*, $C_6H_2(OH)_3 \cdot COOMe$ [COOMe : $(OH)_3$ = 1 : 3 : 4 : 5], crystallises from ether, melts at 192° with evolution of gas, and is readily soluble in water and alcohol.

Methyl trimethylgallate, $C_6H_2(OMe)_3 \cdot COOMe$, obtained by heating the preceding compound with methyl iodide, methyl alcohol, and potash, is a colourless, crystalline compound which melts at 81° and boils at 274—275°.

Trimethylgallic acid, $C_6H_2(OMe)_3 \cdot COOH$, crystallises from ether or water in needles and melts at 167°.

Methyl pyrogallocarboxylate, $C_6H_2(OH)_3 \cdot COOMe$ [COOMe : $(OH)_3$ = 1 : 2 : 3 : 4], crystallises from water in long, shining, colourless needles which lose their water ($2\frac{1}{2}$ mols.) below 100°, and melt at 151—152°.

Methyl trimethylpyrogallocarboxylate, $C_6H_2(OMe)_3 \cdot COOMe$, is a colourless oil, boils at 281°, and is insoluble in water and dilute alkalis. *Trimethyl pyrogallocarboxylic acid*, $C_6H_2(OMe)_3 \cdot COOH$, separates from ether in shining crystals melting at 99°.

Propyltrimethylpyrogallol, $C_6H_2(C_3H_7)(OMe)_3$, prepared by treating propylpyrogallol with methyl iodide, methyl alcohol, and potash, is a colourless oil, boils at 164°, and is readily soluble in alcohol and ether, but insoluble in water. When warmed with concentrated nitric acid, it yields dimethoxyquinone melting at 149° (?), and when boiled with potassium permanganate it yields trimethylgallic acid.

The dimethoxyquinone formed by the action of concentrated nitric acid on trimethylpyrogallol is therefore a derivative of tetrhydroxybenzene [$(OH)_4$ = 1 : 3 : 4 : 5]. F. S. K.

Gallic Acid and Tannin. By C. Böttinger (*Annalen*, **246**, 124—128).—When a solution of the purest commercial tannin is boiled with sodium acetate and phenylhydrazine hydrochloride, a yellow coloration is produced which is followed by a precipitate. This is due to the presence of a small quantity of an impurity which closely resembles tannin, and is absorbed by hide. W. C. W.

Cocatannic Acid. By C. J. H. Warden (*Pharm. J. Trans.*, **18**, 985—987).—From the leaves of *Erthyroxylon coca*, grown in India, the author extracted cocatannin, $C_{17}H_{22}O_{10}$, by treatment with alcohol containing one-sixtieth of sulphuric acid. The substance, after purification, presented itself as a mass of minute filiform crystals of a yellow colour, without taste or odour, and soluble in water, alcohol, or ether. The hot aqueous solution has an acid reaction, and gives no coloration with ferrous salts, but with ferric salts a greenish coloration. The solid acid is easily soluble in alkalis, yielding yellow solutions. The aqueous solution does not precipitate gelatin. The acid appears to be bibasic, and closely allied to quercitrin.

R. R.

Hydrophthalic Acid. By A. v. Baeyer (*Ber.*, **21**, 2271—2272).—The author is investigating the hydrophthalic acids, and has found that Graebe and Born's hydrophthalic acid is a mixture of several

substances. A crystalline dihydro-acid was obtained which yields a mixture of several tetrahydro-acids when boiled with sodium amalgam and water. These acids are very readily transformed, to some extent, into isomeric compounds. Terephthalic acid also exists in forms which very readily undergo intramolecular change, so that the hydro-derivatives of benzenedicarboxylic acids exist in two kinds of mutually convertible isomeric forms, the one depending on the geometric position of the carboxyl-groups, the other on the position of the double binding. F. S. K.

Dichloroterephthalic Acid and Dichlorodihydroterephthalic Acid. By S. Levy and A. Andreocci (*Ber.*, **21**, 1959—1964; compare this vol., p. 840).—Paradichloroterephthalic acid, purified by sublimation, melts at 305—306°.

Barium paradichloroterephthalate, $(C_8H_2Cl_2O_4)Ba + 4H_2O$, crystallises in slender needles, and is very readily soluble in water. The *silver* salt, $C_6H_2Cl_2(COOAg)_2$, is insoluble in water, and becomes rose-coloured when exposed to light.

Methyl paradichloroterephthalate, $C_6H_2Cl_2(COOMe)_2$, crystallises from methyl alcohol in colourless, nacreous plates melting at 131—132°.

Nitroparadichloroterephthalic acid, $NO_2{\cdot}C_6HCl_2(COOH)_2$, is obtained in the preparation of dichloroterephthalic acid (*loc. cit.*). It crystallises from water in yellowish needles, and is readily soluble in alcohol and ether. When heated, it becomes more intensely yellow at about 180°, turns brownish-black at about 205°, and melts, with rapid evolution of gas, at 225—226°. The *ammonium* salt,

$$NO_2{\cdot}C_6HCl_2(COONH_4)_2,$$

crystallises from water, in which it is very readily soluble, in orange-red needles. The *calcium* salt crystallises in orange-red needles, and is rather easily soluble in water.

Methyl nitroparadichloroterephthalate, $NO_2{\cdot}C_6HCl_2(COOMe)_2$, crystallises from dilute methyl alcohol in small, colourless, shining leaves, and melts at 207—208° to a brownish-red liquid, which is decomposed on further heating.

Paradichlorodihydroterephthalic acid (*loc. cit.*) is oxidised at once by alkaline permanganate, and yields dihydroterephthalic acid when treated with sodium amalgam. F. S. K.

The Reduction-products of Terephthalic Acid. By A. v. Baeyer (*Annalen*, **245**, 138—190; see this vol., p. 1069).

Action of Ethyl Sodiomalonate on Tribromodinitrobenzene. By C. L. Jackson and W. S. Robinson (*Ber.*, **21**, 2034—2035).—*Ethyl bromodinitrophenylmalonate*, $(NO_2)_2C_6H_2Br{\cdot}CH(COOEt)_2$, is prepared by adding an alcoholic solution of ethyl sodiomalonate to tribromodinitrobenzene dissolved in ether. After two to three hours, the whole is filtered, treated with dilute sulphuric acid, again filtered, and heated on a water-bath to get rid of the ether; on cooling, the residue

deposits crystals of the new compound. It forms flat, pale-yellow needles, melts at 75°, and dissolves readily in the usual solvents except water, in which it is insoluble. The *sodium-derivative*,

$$C_6H_2Br(NO_2)_2{\cdot}CNa(COOEt)_2,$$

is red, and dissolves in water, alcohol, and ether.

Tribromodinitrobenzene also reacts with ethyl sodacetoacetate.

N. H. M.

Reissert's Pyranilpyroïnlactone, Pyranilpyroic and Anilsuccinic Acids. By R. Anschütz (*Annalen*, **246**, 115—123).—The anilosuccinic acid which Reissert (this vol., p. 694) obtained by the oxidation of pyranilpyroic (mesaconilic) acid is in reality oxanilacetic acid. The dihydropyranilpyroic acid and dihydropyranilpyrolactone derived from pyranilpyroic acid are identical with the anilide and anilic acid of pyrotartaric acid. W. C. W.

Phenoldicarboxylic Acids. By E. Lippmann and F. Fleissner (*Monatsh.*, **9**, 296—307).—When orthohydroxyquinoline (80 grams), potassium xanthate (90 grams), and sufficient absolute alcohol to form a stiff paste, are heated for 24 hours in a water-bath, the mixture becomes red, and solidifies on cooling to a hard mass from which *hydroxyquinolinecarbodithionic acid*, $HO{\cdot}C_6H_9N{\cdot}CSSH$, may be readily isolated. It forms very small red crystals which melt with decomposition at 180°, is only very sparingly soluble in water, alcohol, ether, benzene, and carbon bisulphide, but dissolves readily in alkalis, forming a red solution. The ammonium and barium salts are both only sparingly soluble in water. On oxidising the acid with alkaline permanganate, quinolinic acid melting at 231° is formed; hence the CSSH-group is attached to the benzene and not to the pyridine ring of the nucleus.

When the dithio-acid is heated with mercuric chloride, it loses sulphur and forms a new *hydroxyquinolinecarboxylic acid* crystallising in slender, lustrous needles, containing 1 mol. of water, and melting at 256° with liberation of carbonic acid and formation of orthohydroxyquinoline. The acid is more soluble in water than in alcohol, and only traces of it are dissolved by ether, benzene, and chloroform. Ferric chloride colours its aqueous solution a deep brownish-red, and ferrous sulphate in concentrated solutions precipitates the ferrous salt in the form of a brown crystalline mass. The hydrochloride crystallises in small, white needles containing $2\frac{1}{2}$ mols. of water. The potassium salt forms very pale-yellow needles, the barium, silver, and mercury salts are not characteristic, and the platinochloride is so unstable as to be readily decomposed by water.

Resorcinolcarbodithionic acid, $C_6H_3(OH)_2{\cdot}CSSH$, is obtained by heating a mixture of absolute alcohol and equivalent parts of potassium xanthate and resorcinol. It forms bright yellow needles melting at 131°, and dissolves in hot water with partial decomposition and evolution of hydrogen sulphide. It does not lose its sulphur when heated with mercuric chloride, but potash readily converts it into what seems to be a mixture of two isomeric dihydroxybenzoic

acids (resorcylic acids) which decomposes at 200°, but when heated in sealed tubes melts at 216°. Ferric chloride turns the aqueous solution red, but the colour disappears on adding excess of soda.

G. T. M.

Anilinetrisulphonic Acid. By G. T. Hartshorn and C. L. Jackson (*Ber.*, **21**, 2032—2033).—*Anilinetrisulphonic acid*,

$$NH_2{\cdot}C_6H_2(SO_3H)_3,$$

is prepared by heating potassium β-anilinemetadisulphonate with sulphuric acid, or aniline with an excess of sulphuric acid and the calculated amount of potassium sulphate, in a dish until the mixture begins to carbonise. It crystallises from water in flat, lustrous prisms readily soluble in hot water; when heated, it blackens and decrepitates but does not melt. The *dipotassium salt* (with 3 mols. H_2O) crystallises in thin needles; the *barium salt*, $NH_2{\cdot}C_6H_2{\cdot}SO_3H(S_2O_6Ba) + 3\frac{1}{2}H_2O$, and the *lead salt*, which forms silky needles, are described.

N. H. M.

Phenacyl Compounds. By W. Staedel (*Ber.*, **21**, 2196—2199).—Indoles can be easily obtained from phenacylanilides. When methylphenacylanilide is warmed with zinc chloride, a compound $C_{15}H_{13}N$—probably methylphenylindole—is obtained. It crystallises in shining prisms or plates, melts at 101—102°, and is readily soluble in alcohol or benzene, but only sparingly in light petroleum. It distils without decomposition, and its alcoholic solution colours pine-wood moistened with hydrochloric acid dark red. When heated with methyl iodide and methyl alcohol, it yields a solid compound which behaves like a dihydroquinoline. The *picrate* crystallises in red needles. Methylphenacylanilide is completely decomposed when boiled with aniline for several hours with formation of methylaniline and a crystalline compound, the properties of which agree with those of 2-phenylindole.

F. S. K.

Aldine Formation. By E. Braun and V. Meyer (*Ber.*, **21**, 1947—1949).—When esoamidoacetophenone hydrochloride is dissolved in water free from air, precipitated with ammonia free from air, and the precipitate, after keeping for some time, washed in an atmosphere of hydrogen and then without drying dissolved in hydrochloric acid, a solution of the original hydrochloride is obtained. But if the precipitate is dried in an atmosphere of hydrogen at 110°, it becomes to a great extent insoluble in hydrochloric acid, and after crystallising from boiling alcohol it yields a considerable quantity of isoindole (compare this vol., p. 700). If this same precipitate is washed in absence of air, and dried in a partial vacuum, a crystalline powder is obtained which is partly soluble in hydrochloric acid, and contains 11·9 per cent. of nitrogen.

When isonitrosomethyl ethyl ketone is reduced with stannous chloride and hydrochloric acid, a solution is obtained which does not contain tetramethylaldine (*loc. cit.*). If the solution is freed from tin by means of hydrogen sulphide, evaporated, and the residue extracted with alcoholic ether, a deliquescent salt is obtained. This salt forms a platinochloride which is totally different from the

platinochloride of tetramethylaldine, and it yields tetramethylaldine when treated with alkali.

A base is formed when benzil monoxime is reduced with stannous chloride and hydrochloric acid; it is a colourless crystalline compound, insoluble in ether, &c., but readily soluble in dilute hydrochloric acid, and different in all respects from tetraphenylaldine. It contains 6·46 per cent. of nitrogen. A solution of its hydrochloride can be evaporated without undergoing change (*loc. cit.*). F. S. K.

Orthodinitrostilbene. By C. A. Bischoff (*Ber.*, **21**, 2071—2078). —*Orthodinitrostilbene*, $C_{14}H_{10}N_2O_4$, is prepared by heating a solution of 17 grams of orthonitrobenzyl chloride in 50 grams of warm 98 per cent. alcohol with a solution of 5·6 grams of potash in 56 grams of alcohol. When cold, the crystals are washed with 90 per cent. alcohol. It is then several times extracted with boiling glacial acetic acid, and the insoluble portion crystallised from chloroform, from which it separates in long, bright yellow needles melting at 196°. The acetic acid solution deposits crystals of varying melting point; in precipitating the last mother-liquor with water, and repeatedly crystallising the precipitate from glacial acetic acid, washing with alcohol and water, short, slightly grey needles are obtained which melt at 126°. Both compounds are orthodinitrostilbene; to the former (m. p. 196°), which is the chief product, the name of transorthodinitrostilbene is ascribed, and to the latter, cis-orthodinitrostilbene. This takes up bromine without evolution of hydrogen bromide, yielding a bromo-compound melting at 215°.

Transorthodinitrostilbene is insoluble in water, very sparingly soluble in alcohol, ether, and light petroleum, more soluble in benzene and carbon bisulphide; when treated with bromine, it yields the *dibromide*, $C_{14}H_{10}N_2O_4Br_2$. This crystallises in thin, yellowish plates resembling bismuth nitrate, melts at 226°, and dissolves sparingly in hot acetone and ethyl acetate; it is more sparingly soluble than the bromine-derivative of the cis-compound.

Attempts were made to obtain a reaction between ethyl sodiochloromalonate and orthodinitrostilbene dibromide analogous to that described by Conrad (Abstr., 1881, 168) between the former compound and benzyl chloride, but without success.

Orthonitramidostilbene, $C_{14}H_{12}N_2O_2$, is obtained by heating the dinitro-compound with alcoholic ammonium sulphide for 10 hours at 100°. It is amorphous, and has no definite melting point, and is soluble in ether and alcohol. N. H. M.

Azo-dyes from Orthodiamidostilbene. By C. A. Bischoff (*Ber.*, **21**, 2078—2084).—*Orthodiamidostilbene*, $C_{14}H_{14}N_2$, is prepared by treating an intimate mixture of the dinitro-derivative and granulated tin with hydrochloric acid (sp. gr. 1·19), and after half an hour heating the mixture on a water-bath; the temperature rises suddenly to 110°. In 10 minutes, the reaction is complete, and on cooling the stannochloride separates. The free base crystallises in large, thin prisms of a golden lustre and melts at 176°; the alcoholic solution shows violet-blue fluorescence. The *hydrochloride* forms concentrically

grouped needles, sparingly soluble in water; the *sulphate* crystallises in long prisms; the *platinochloride* is a yellow powder. The base is diazotised by treating 16·8 grams with 40 c.c. of hydrochloric acid (sp. gr. = 1·145), and diluting to 2 litres. 250 c.c. of this solution is diazotised at a time with the corresponding amount of nitrite.

The tetrazo-compound was treated in the usual manner with—(1) Salicylic acid; (2) Bayer's sodium naphtholsulphonate; (3) Schaeffer's sodium naphtholsulphonate; (4) α-naphthylaminesulphonic acid; (5) sodium naphtholdisulphonate (R. salt), each 2 mols. The diazo-compound was treated with the following compounds, and then further diazotised—(6) 1 mol. of α-naphthylaminesulphonic acid and 1 mol. resorcinol; (7) 1 mol. of R. salt and 1 mol. of α-naphthylaminesulphonic acid; (8) 1 mol. of R. salt and 1 mol. of phenol. The compound obtained under (4), $C_2H_2[C_6H_4{\cdot}N_2{\cdot}C_{10}H_5{\cdot}NH_2(SO_3H)]_2$, was converted into a tetrazo-derivative, treated with 2 mols. of α-naphthylaminesulphonic acid, again tetrazotised and treated with 2 mols. of α-naphthylaminesulphonic acid; in this way, the compound $C_2H_2[C_6H_4{\cdot}N_2{\cdot}C_{10}H_5(SO_3H){\cdot}N_2{\cdot}C_{10}H_5(SO_3H){\cdot}N_2{\cdot}C_{10}H_5(SO_3H){\cdot}NH_2]_2$ is obtained.

Tables are given showing the properties and reactions of the dyes.

N. H. M.

Action of Heated Copper on a Mixture of the Vapours of Phenol and Carbon Bisulphide. By T. Carnelley and J. Dunn (*Ber.*, **21**, 2005—2009).—*Diphenylene ketone*, isomeric with ordinary diphenylene ketone, is formed when phenol and carbon bisulphide are passed over heated copper, but the greater portion of the phenol is undecomposed. The ketone crystallises from alcohol in white needles or leaves, melts at 83°, is sparingly soluble in cold, more readily in hot alcohol. It is readily soluble in glacial acetic acid, insoluble in water, is not oxidised when boiled with dilute nitric acid, and is extremely stable towards oxidising agents.

The *bromine*-derivative, $C_{13}H_7BrO$, prepared by treating a carbon bisulphide solution of the ketone with bromine, crystallises from alcohol in shining scales melting at 104°.

The *nitro*-derivative, prepared by heating the ketone with strong nitric acid at 170°, melts at 220—230°, and is insoluble in alcohol and water.

When the ketone is reduced, a compound is obtained, which crystallises from alcohol in angular plates, melts at 79—80°, and is moderately soluble in hot, but only sparingly in cold alcohol. The composition of this compound is probably $C_{45}H_{30}O_2$, or $C_{45}H_{32}O_2$.

F. S. K.

Benzilorthocarboxylic Acid. By C. Graebe and P. Juillard (*Ber.*, **21**, 2003—2005).—*Benzilorthocarboxylic acid*,

$$COPh{\cdot}CO{\cdot}C_6H_4{\cdot}COOH,$$

is formed when desoxybenzoïncarboxylic acid is oxidised with alkaline potassium permanganate. The reaction-product crystallises from hot water or dilute alcohol, either in dazzling white, or in pale-yellow

crystals, and sometimes both varieties occur together. The yellow colour is not due to any impurity; when the white crystals are heated at 115—125°, they are converted into the yellow variety. Both modifications melt at 141·5°, and are anhydrous. The white crystals are obtained when the yellow ones are dissolved in alkali and precipitated with acids.

Benzhydroldicarboxylic acid, $COOH{\cdot}CPh(OH){\cdot}C_6H_4{\cdot}COOH$, is formed when benzilorthocarboxylic acid is warmed with alkalis. It is readily soluble in alcohol, benzene, and ether, sparingly soluble in water, and is converted into phenyl phthalide when heated. It has no definite melting point; when placed in a bath at 80—90° it melts, and is at once decomposed. The *potassium* salt crystallises with 2 mols. H_2O. F. S. K.

Action of Citraconic Acid on Naphthylamine. By T. Morawski and M. Gläser (*Monatsh.*, **9**, 284—295).—*α-Naphthylamine citraconate* is formed when solutions of α-naphthylamine and citraconic acid dissolved in benzene are brought together in molecular proportions. It forms colourless needles, which turn yellow on exposure to light, melt at 99°, and decompose at 100°.

β-Naphthylamine citraconate is formed when alcoholic solutions of β-naphthylamine and citraconic acid are mixed in molecular proportions and allowed to evaporate at low temperatures. It crystallises in pale-yellow needles, which melt at 173—174°, with loss of water.

Citracon-α-naphthil, $C_{10}H_7{\cdot}N{:}C_5H_4O_2$, is most conveniently prepared by heating together molecular proportions of citraconic acid and α-naphthylamine in sealed tubes at 140—150° for half an hour. Crystallised from hot alcohol, it forms beautiful yellow plates, which melt at 142—143°, and boil at 360° with partial decomposition. The corresponding β-naphthylamine compound is prepared in a similar way, and much resembles its isomeride. It crystallises from most of the ordinary solvents in very small needles.

On brominating 1 mol. of the α-compound dissolved in acetic acid with 2 mols. of bromine, the dibromo-derivative, $\alpha\text{-}C_{10}H_6Br{\cdot}N{:}C_5H_3BrO_2$, is formed; this crystallises in pale-yellow prisms, and melts at 199°. The corresponding dibromo-β-derivative forms yellow needles melting at 181°. G. T. M.

Action of Ammonium Sulphide on some Dinitro-compounds. By E. Heim (*Ber.*, **21**, 2301—2309).—*Dinitrophenyl-α-naphthylamine* is obtained by heating bromodinitrobenzene (1 mol.) with α-naphthylamine (1 mol.) in alcoholic solution (compare Heim, this vol., p. 488). It crystallises from glacial acetic acid or absolute alcohol in orange-red needles, melts at 190·5°, is very readily soluble in benzene, toluene, acetone, and chloroform, but insoluble in water. It dissolves in alcoholic potash, yielding a dark-red, and in concentrated sulphuric acid, forming a dark-blue solution.

Nitroamidophenyl-α-naphthylamine, prepared by reducing the preceding compound with ammonium sulphide in alcoholic solution, crystallises from alcohol or acetic acid in dark-yellow needles, melts at 145—147°, and is very readily soluble in alcohol, glacial acetic acid,

chloroform, warm benzene, and toluene, moderately soluble in ether, and sparingly in hot water. It dissolves in concentrated sulphuric acid, forming a dark blue solution, from which water precipitates brown flocks. The amido-group is in the ortho-position to the imido-group.

Nitrazoimidophenyl-α-naphthylamine, $C_{10}H_7 \cdot N < \overset{C_6H_3(NO_2)}{N=\!=\!=N} >$, is obtained by treating an alcoholic solution of the preceding compound with hydrochloric acid and ethyl nitrite. It crystallises from hot absolute alcohol, in which it is only sparingly soluble, in cinnamon-brown, shining needles, melts at 182°, is readily soluble in acetone and chloroform, moderately soluble in benzene, toluene, and hot glacial acetic acid, sparingly in ether and carbon bisulphide, and insoluble in water. It dissolves in concentrated sulphuric acid, forming a dark-green solution, from which it is precipitated unchanged by water, as is also the case with the solution in hot alcoholic potash.

Nitrophenophenanthrazine, $C_{20}H_{11}N_3O_2$, is obtained by heating nitrophenyleneorthodiamine with a glacial acetic acid solution of phenanthraquinone. It melts at 251°, and at a higher temperature sublimes in slender, yellow needles, with only slight decomposition. It is insoluble in water, very sparingly soluble in alcohol, ether, and glacial acetic acid, moderately soluble in acetone, and readily in chloroform, and boiling benzene or toluene. It gives a purple-red solution with concentrated sulphuric acid, but on adding water the colour changes to orange, and finally yellow flocks are precipitated.

Amidophenophenanthrazine, melting at 179°, is identical with the compound obtained by Witt (Trans., 1886, 401) from phenanthraquinone and a solution of reduced chrysoïdine.

Nitroethenylorthophenylenediamine, $C_6H_3(NO_2) < \overset{-N}{NH} > CMe$, is obtained when nitro-orthophenylenediamine is heated for four hours at 190° with acetic anhydride. It crystallises in yellowish-brown, well-defined needles containing water, which is given off at 120°. It melts at 216°, is readily soluble in alcohol, benzene, chloroform, acids, and hot water, and moderately soluble in ether.

Nitroamidodimethylaniline, $C_8H_{11}N_3O_2$, prepared by reducing dinitrodimethylaniline with alcoholic ammonium sulphide, crystallises from boiling water in orange-yellow needles, melts at 63°, and is sparingly soluble in hot water, but readily soluble in ordinary organic solvents, and in dilute mineral acids. It distils in small quantities with steam or alcohol vapour, but soon decomposes in aqueous or alcoholic solution. It is not changed by heating with alcoholic potash, and yields a crystalline *acetyl*-derivative. Paranitrodimethylaniline is obtained when an alcoholic solution of the preceding compound is treated with hydrochloric acid and ethyl nitrite. F. S. K.

Phenylhydrazine-derivatives of Hydroxynaphthaquinone. By T. Zincke and H. Thelen (*Ber.*, **21**, 2200—2205).—*Benzaldishydroxynaphthaquinone*, $CHPh(C_{10}H_4O_2 \cdot OH)_2[OH : CHPh = 2 : 3]$, is readily obtained by heating 1 part of hydroxynaphthaquinone with 1 part of benzaldehyde and 2—3 parts of alcohol at 100° for some

hours. It crystallises from dilute acetic acid, or from a mixture of benzene and light petroleum in small, bright-yellow needles, melts at 211—214°, and is readily soluble in alcohol, glacial acetic acid, ether, chloroform, and benzene, but almost insoluble in light petroleum. When warmed with phenylhydrazine in alcoholic solution, it yields the same compound ($C_{39}H_{28}N_4O_4$) as is obtained from hydroxynaphthaquinone hydrazide and benzaldehyde (Abstr., 1884, 1359).

Ethylidenedishydroxynaphthaquinonehydrazide,

$$CHMe[C_{10}H_4O(N_2HPh)\cdot OH]_2,$$

prepared by heating an alcoholic solution of hydroxynaphthaquinonehydrazide with acetaldehyde, crystallises from chloroform or aniline in small, dark-red needles, melts at 258° with decomposition, and is sparingly soluble in hot benzene or hot acetic acid, but almost insoluble in alcohol.

Acetone and hydroxynaphthaquinonehydrazide yield a condensation product, which crystallises from chloroform in reddish-yellow needles; it melts at 245—250°, and is readily soluble in alcoholic, but insoluble in aqueous alkali. F. S. K.

Terpenes. By O. Wallach (*Annalen*, **245**, 241—278; compare this vol., p. 60).—*Pinene nitrosochloride*, $C_{10}H_{16},NOCl$, is prepared by mixing 14 c.c. of pinene, 20 c.c. of amyl nitrite, and 34 c.c. of glacial acetic acid; to each 6 c.c. of this mixture, which is well cooled, 3 c.c. of a mixture of equal volumes of hydrochloric (33 per cent.) and acetic acids is gradually added, so that the blue colour produced disappears each time before the addition of fresh acid. The *nitrosobromide*, $C_{10}H_{16},NOBr$, is obtained by very gradually adding 7 c.c. of 60 per cent. hydrobromic acid diluted with 10 c.c. of alcohol to a well-cooled mixture of 14 c.c. of oil of turpentine, 20 c.c. of amyl nitrite, and 20 c.c. of alcohol. It melts at 91—92°, and resembles the nitrosochloride. *Pinene nitrolpiperidine*, $C_{10}H_{16}NO\cdot C_5NH_{10}$, prepared by heating the nitrosochloride with an excess of pure piperidine, melts at 118—119°; the *hydrochloride* is sparingly soluble. Camphene could not be made to combine with nitrosyl chloride.

Limonene nitrosochloride is also readily prepared by the method given for the pinene-derivative; the *hydrochloride*, $C_{10}H_{16},NOCl,HCl$, melts at 113—114°. *Limonene nitrosobromide*, $C_{10}H_{16},NOBr$, melts at 90·5°. *Limonene nitrosate*, $C_{10}H_{16},NO(ONO_2)$, is formed when a mixture of equal volumes of limonene and amyl nitrite is cooled and treated with ½ vol. of glacial acetic, 1 mol. of nitric acid (sp. gr. = 1·395); it was not purified or analysed. *Limonene hydrochloride*, $C_{10}H_{17}Cl$, is prepared by adding 100 c.c. of limonene to a mixture of 100 c.c. of fuming hydrochloric acid (38 per cent.) and 100 c.c. of glacial acetic acid, heating the whole for some hours at 70°, pouring it into water, and distilling in a vacuum. It is dextrorotatory. Sp. gr. = 0·98°. Mol. refraction = 49·86. It boils at 90° in a vacuum, but when kept the boiling point is much higher. *Hydrochlorolimonene nitrosate*, $C_{10}H_{17}Cl,NO(ONO_2)$, is obtained by the action of 60 per cent. nitric acid on a mixture of limonene chloride and amyl nitrite; it is identical with the compound obtained by Maissen from

limonene (*loc. cit.*). The *nitrosochloride*, $C_{10}H_{17}Cl,NOCl$, prepared from the chloride in a manner similar to the nitrosochloride from limonene, melts at 109°. *Hydrochlorolimonene nitrolanilide*,

$$C_{10}H_{12}Cl\cdot NO\cdot NHPh,$$

is formed when 13 grams of the nitrosate, $C_{10}H_{17}Cl,N_2O_4$, is treated with 9 grams of aniline and 40 c.c. of alcohol; it melts at 135—136°. *Hydrochlorolimonene nitrolparatoluidide*, $C_{10}H_{17}Cl,NO\cdot NH\cdot C_6H_4Me$, prepared like the anilide, crystallises with 1 mol. EtOH in well-formed octahedra, melting at 135°; when free from alcohol, it melts at 145—146°. When a mixture of 13 grams of the hydrochloronitrosate, 6 grams of dimethylaniline, and 50 c.c. of alcohol is heated, the compound $C_{10}H_{17}Cl,NO\cdot OEt$ is formed. It separates from its alcoholic solution in well-formed crystals melting at 144—145°. If methyl alcohol is used instead of ethyl alcohol, the compound $C_{10}H_{17}Cl,NO\cdot OMe$, crystallising in well-formed prisms melting at 139° is obtained. All the derivatives obtained from the hydrochloronitrosate yield non-chlorinated compounds when boiled with alcoholic potash.

Dipentene dichloride is readily prepared by passing hydrogen chloride over (not into) dipentene diluted with $\frac{1}{2}$ vol. of glacial acetic acid. The *nitrosochloride*, $C_{10}H_{16},NOCl$, is obtained by shaking a well-cooled mixture of 6 c.c. of pure dipentene, 6 c.c. of methyl alcohol, and 9 c.c. of amyl nitrite with 7 c.c. of fuming hydrochloric acid, which is gradually added. It melts at 101—102°, and is optically inactive. It is converted by alcoholic potash into the compound $C_{10}H_{15}NO$ (isomeric with carvoxime), which melts at 92—93°, dissolves very sparingly in boiling water, more readily in hot alcohol, and is optically active. Goldschmidt and Kisser obtained (Abstr., 1887, 925) by the action of dilute sulphuric acid on isocarvoxime a compound of the formula $C_{10}H_{15}NO$, melting at 94°. *Dipentene nitrolpiperidine*, $C_{10}H_{16},NO\cdot C_5NH_{10}$, crystallises from alcohol in colourless needles melting at 153—154°. The *nitrosate*, $C_{10}H_{16},NO(ONO_2)$, is prepared by adding 3·5 grams of nitric acid (sp. gr. = 1·395) to 5 grams of dipentene, 4 grams of amyl nitrite, and 2 c.c. of glacial acetic acid previously cooled. Alcohol is added until a clear solution is formed, and then water until just turbid. It is then left for some hours in a freezing mixture. It crystallises in lozenge-shaped plates, melts at 84° with decomposition, and dissolves very readily in benzene. When heated with alcoholic potash, the nitroso-compound melting at 93° is formed. The *hydrochloride*, $C_{10}H_{16},N_2O_4,HCl$, was prepared.

Sylvestrene nitrosochloride, $C_{10}H_{16},NOCl$, melts at 106—107°, and dissolves very readily in chloroform. It is strongly dextrorotatory.

Terpinene benzoyl isonitrosite, $C_{10}H_{15}(ONO),NO\cdot COPh$, is prepared by adding 20 grams of benzoic chloride to 30 grams of terpinene nitrosite, and 300 c.c. of dry ether, and keeping the whole in a closed vessel for some days. It crystallises from alcohol in plates melting at 77—78°. It is probable that terpinene nitrosite is a nitroso-compound, $NO\cdot C_{10}H_{16}\cdot ONO$, and that in presence of reagents it has a tendency to go over into an isonitroso-compound. N. H. M.

Cheken Leaves. By F. Weiss (*Pharm. J. Trans.*, **18**, 1051—1053).—The most important constituent of cheken leaves (*Myrtus cheken*) is an ethereal oil of greenish-yellow colour and pleasant odour, which on slow evaporation leaves 5 per cent. of a resinous matter. The oil is not changed by cold, and is miscible with ether, chloroform, and absolute alcohol in all proportions. Its specific gravity is 0·8975 at 15°, and polarisation $[\alpha]_j = +23·5°$. Its chief constituent is a fluorescent hydrocarbon, $C_{10}H_{16}$; boiling point, 156° (uncorr.); sp. gr. = 0·8635 at 15°; rotatory polarisation, $[\alpha]_j = 31·28°$, identical with Wallach's pinene. A second portion containing oxygen yields with bromine brick-red crystals of cineol dibromide (Wallach), and is therefore cineol. The third portion is a dense yellow oil, smelling strongly of cheken. From the leaves, after separation of the ethereal oil, the author obtained by extraction with alcohol, &c., several principles, which, after isolation by processes described in the paper, he examined, and has described as *chekenone*, $C_{40}H_{44}O_8$, an indifferent substance, melting at 203—204°, and resisting ordinary reagents; *chekenic acid*, $C_{12}H_{11}O_3$, melting at 224—225°, and forming yellow, easily decomposed compounds with bases; *cheken bitter*, an intensely bitter substance, with unpleasant odour, but without toxic properties; *chekenetin*, yellow-green, acicular crystals, $C_{11}H_7O_6 + H_2O$, which form bright green-coloured solutions with alkalis, changing to blue-violet, and finally to onion-peel red. This colouring principle is, the author believes, allied to the quercetin-group. R. R.

Apiole. By G. Ciamician and P. Silber (*Ber.*, **21**, 2129—2133).—Apiolaldehyde (compare this vol., p. 847) is best obtained from isapiole by treating the latter, in a glacial acetic acid solution, with chromic acid, the yield being 35 to 40 per cent.

Acetyl apiolaldoxime, $C_9H_9O_4(CH{:}NO{\cdot}COMe)$ (*loc. cit.*), crystallises from alcohol in long, shining prisms, melts at 128—129°; it is soluble in hot alcohol and ether, but only sparingly soluble in hot, and almost insoluble in cold, water. It gives the same colour reaction with sulphuric acid as apiolaldoxime (*loc. cit.*).

Bibromapione, $C_9H_8Br_2O_4$, prepared by warming a glacial acetic acid solution of apiolic acid or apiolaldehyde with an excess of bromine, crystallises from alcohol in prisms or needles, melts at 99—100°, and gives the sulphuric acid reaction. It is readily soluble in ether, ethyl acetate, warm alcohol, and glacial acetic acid, but sparingly soluble in warm, and almost insoluble in cold water.

From its behaviour when heated with hydriodic acid, apiolic acid probably contains two methoxy-groups.

A *nitro*-compound, probably $C_8H_6N_2O_7$, is formed when apiolic acid is nitrated; it crystallises in long, flat, yellow needles, melts at 118°, and is soluble in ether, alcohol, and acetic acid, but insoluble in water and aqueous alkalis. F. S. K.

Cubebin. By C. Pomeranz (*Monatsh.*, **9**, 323—326; compare this vol., p. 162).—When cubebin is oxidised with permanganate in alkaline solution, it yields piperonylic acid, and may therefore be regarded as a compound derived from the group $CH_2.O_2{:}C_6H_4$, in which one of

the atoms of hydrogen in the benzene nucleus is replaced by the group C_4H_5O. By the action of benzoic chloride on cubebin, hydrogen chloride is liberated, and an atom of hydrogen in the C_3H_5O-group is displaced by benzoyl, hence the oxygen is present in the form of hydroxyl. From the benzoyl-derivative, benzoic acid may be readily regenerated by hydrolysis. Cubebin, therefore, has the constitution $H_2C{:}O_2{:}C_6H_3{\cdot}C_3H_4{\cdot}OH$ [$O_2 : C_3H_5O = 1 : 2 : 4$]. G. T. M.

Organic Compound which Hydrogenises Sulphur in the Cold. By J. DE REY-PAILHADE (*Compt. rend.*, **106**, 1683—1684).—Dried yeast suspended in its own volume of alcohol of 86°, and agitated frequently for two days in a closed flask, yields a limpid yellowish liquid with an acid reaction. If sulphur, moistened with alcohol, is introduced into this solution, hydrogen sulphide is evolved. When yeast, triturated with sulphur, is introduced into a solution of sugar, carbonic anhydride and hydrogen sulphide are evolved, as Dumas previously observed.

The action of this substance is greater at 35—40° than at the ordinary temperature, but if the alcoholic solution is heated to 70° and cooled, it becomes turbid and loses its power of producing hydrogen sulphide. The same change takes place slowly at the ordinary temperature. The power of hydrogenising sulphur also gradually disappears if fresh yeast is suspended in solutions of acids or alkalis, or is triturated with neutral salts. Water or ether, agitated with fresh yeast, acquires no such power.

Fresh animal tissues, macerated with their own weight of alcohol of 86°, yield a solution which gives no hydrogen sulphide with sulphur. If, however, the residue is triturated with sulphur, hydrogen sulphur is given off in considerable quantity. It follows that the substance, to which the author gives the name *philothion*, exists in animal tissues in a form different from that in which it exists in yeast. It stands to sulphur in the same physiological relation as hæmoglobin to oxygen, that is to say, it renders it soluble and assimilable.

C. H. B.

Philothion. By J. DE REY-PAILHADE (*Compt. rend.*, **107**, 43—44).—Philothion (preceding Abstract) is formed in the life processes of yeast, and when the latter is treated with various reagents, its death always precedes the destruction of the philothion. Concentrated methyl alcohol readily extracts it from yeast. A current of air passed through an alcoholic solution for two hours causes no appreciable decomposition of philothion.

Philothion exists in white of egg and in fresh sheep's blood, but not in lamb's bile or human urine. It occurs to some extent in plants, and is found, for example, in the tips of young shoots of asparagus. It does not exist, however, in the young seeds of beans and peas.

C. H. B.

Oxyazoles and their Derivatives. By M. LEWY (*Ber.*, **21** 2192—2196 (compare this vol., p. 55).—*Phenylmethyloxyazole*,

$$<\begin{matrix}N{:}CPh\\CH{:}CMe\end{matrix}>O,$$

is formed when chloracetone is heated with 2 mols. of benzamide for about two hours. It is a colourless oil, boils at 238—241°, turns yellow on exposure to the air, and is soluble in strong acids. The *platinochloride*, $(C_{10}H_9NO)_2,H_2PtCl_6 + 2H_2O$, crystallises from dilute hydrochloric acid in bright yellow, slender needles, melting at 170°.

Methylphenylimidazole, $<^{N:CPh}_{CH:CMe}>NH$, is obtained by heating the preceding compound with an excess of concentrated alcoholic ammonia at 220—230°. It crystallises from aqueous alcohol in long, white needles, melts at 158—159°, and is very readily soluble in strong alcohol or benzene, but only sparingly in ether and cold water. It distils slowly with steam, and when heated alone distils with partial decomposition. The *hydrochloride*, $C_{10}H_{10}N_2,HCl$, crystallises from hot water in long, slender, white needles, which melt at 242° with decomposition. The *platinochloride*, $(C_{10}H_{10}N_2)_2,H_2PtCl_6 + 2H_2O$, crystallises from hot water in large, yellow, flat prisms, melting at 210—212° with decomposition. F. S. K.

Hydantoïns. By A. Pinner (*Ber.*, **21**, 2320—2329).—The metapyrazolone-derivatives described by Pinner and Lifschütz (Abstr., 1887, 1054), must be regarded as derivatives of hydantoïn.

α-Phenylhydantoïn (phenylmetapyrazolone), $<^{CHPh\cdot CO}_{NH\cdot CO-}>NH$, separates in long, white needles, melting at 178°, when 145 grams of benzaldehydecyanhydrin are heated at 100° with 60 grams of carbamide, the product washed with ether, and the residual nitrile warmed with dilute hydrochloric acid for 15 minutes. If, however, the nitrile is heated with concentrated hydrochloric acid for several hours the phenylhydantoïn separates in plates, melting at 178°. Both kinds of crystals retain their specific form when recrystallised from water.

The salt, $C_9H_7KN_2O_2$, separates in small, white needles when the crystalline plates are dissolved in concentrated alcoholic potash (1 mol.) and the solution kept. It is sparingly soluble in alcohol, but readily soluble in water, and the crystalline plates melting at 178° are again obtained when a concentrated aqueous solution is precipitated with hydrochloric acid. It is converted into the potassium salt of pseudophenylhydantoïn when recrystallised from alcohol.

The *potassium* salt of pseudophenylhydantoïn, $C_9H_7N_2O_2K$, separates in slender, white needles, when phenylhydantoïn is boiled with an alcoholic solution of potash (2 mols.); it is almost insoluble in water.

Pseudophenylhydantoïn, $<^{CHPh\cdot CO}_{O\cdot C(NH)}>NH$, is obtained by treating an aqueous solution of the potassium salt with hydrochloric or acetic acid. It is crystalline, insoluble in water and alcohol, and decomposes when heated above 300°, but does not melt.

The conversion of phenylhydantoïn into the pseudo-form does not take place in aqueous, but only in alcoholic solution, and is best carried out very slowly, and at the ordinary temperature, as a little

phenylamidoacetic acid is formed when the solution is heated. Phenylhydantoïn is also partially converted in the pseudo-form when hydrogen chloride is passed into an alcoholic solution for a long time.

Methylphenylhydantoïn, $<^{CHPh\cdot CO}_{NH\cdot CO-}>NMe$, is obtained by heating an absolute methyl alcoholic solution of phenylhydantoïn with potash (1 mol.) and methyl iodide. It crystallises from alcohol in long needles, melts at 161—162°, is sparingly soluble in cold alcohol and dilute alkalis, but readily in hot alcohol.

Ethylphenylhydantoïn, prepared in like manner, separates from water in an oily condition, but soon solidifies to compact, shining prisms melting at 94°. It is readily soluble in alcohol, ether, benzene, and dilute alkalis, moderately soluble in hot, but only sparingly in cold water. It yields barium carbonate, ethylamine, and phenylamidoacetic acid, when heated with an excess of baryta-water, thus proving the constitution assigned to phenylhydantoïn.

Ethylpseudophenylhydantoïn, $<^{CHPh\cdot CO}_{O\cdot C(NH)}>NEt$, separates when a dilute alkaline solution of ethylphenylhydantoïn is kept for some time. It is also obtained by heating a dilute alcoholic solution of pseudophenylhydantoïn with potash (1 mol.) and ethyl bromide. It crystallises in white needles, is sparingly soluble in alcohol, ether, benzene, and boiling amyl alcohol, and insoluble in water. When heated above 300°, it decomposes, but does not melt, and it yields equal quantities of ammonia and ethylamine, together with phenylhydroxyacetic acid, when heated with an excess of baryta-water. This reaction shows that pseudophenylhydantoïn has the constitution given above.

α-Phenylhydantoïc acid, $NH_2\cdot CO\cdot NH\cdot CHPh\cdot COOH$, is obtained when phenylhydantoïn is boiled with baryta-water until the solution commences to become turbid. It crystallises from water in colourless prisms, is sparingly soluble in cold, but readily in hot water, and melts at 178° with evolution of carbonic anhydride and ammonia. When pseudophenylhydantoïn is heated with baryta-water until the solution is distinctly turbid, it yields small quantities of ammonia, carbonic anhydride, and phenylhydroxyacetic acid, but the greater portion remains unchanged.

The *acetyl*-derivative of phenylhydantoïn crystallises from benzene in small needles, melts at 145°, is readily soluble in alcohol, moderately soluble in benzene, sparingly in ether, and insoluble in water. It is readily saponified by soda. F. S. K.

Higher Homologues of the Synthetical Pyridines and Piperidines. By A. Jaccke (*Annalen*, **246**, 32—52).—The ethylic salt of normal propyllutidinehydrodicarboxylic acid,

$$C_5NH_2Me_2Pr^{\alpha}(COOEt)_2,$$

is obtained by adding normal butaldehyde (1 mol.) to a mixture of alcohol and ethyl acetoacetate (2 mols.), in which ammonia (1 mol.) is dissolved. The ethereal salt melts at 118°, crystallises in prisms, and dissolves freely in alcohol and ether. The alcoholic solution

yields *ethyl normal propyllutidinedicarboxylate*, $C_5NMe_2Pr^a(COOEt)_2$, on oxidation with nitrous acid. This substance is an oily liquid, boiling at 308°. The *platinochloride*, $(C_{16}H_{23}NO_4)_2,H_2PtCl_6$, forms orange-coloured prisms and melts at 187°. The free acid,

$$C_5NMe_2Pr^a(COOH)_2 + H_2O,$$

is freely soluble in hot water and melts at 211—212°. The anhydrous acid melts at 247°. When heated with lime, it yields normal *propyllutidine*, $C_5NH_2Me_2Pr$, boiling at 193—196° under a pressure of 718 mm. The hydrochloride and platinochloride are crystalline.

Ethyl hexyllutidinehydrodicarboxylate, prepared by the action of ammonia on ethyl acetoacetate and œnanthol, crystallises in prisms of a yellow colour, and melts at 54°. *Ethyl hexyllutidinedicarboxylate*, $C_6H_{13}\cdot C_5NMe_2(COOEt)_2$, is an oil which crystallises with difficulty. The *platinochloride* forms orange-coloured plates and melts at 141°. The free acid was not obtained in the pure state. *Normal hexyllutidine*, $C_6H_{13}\cdot C_5NH_2Me_2$, boils at 249—251°. The hydrochloride is deliquescent, but the platinochloride crystallises in beautiful prisms melting at 163°. The alcoholic solution of the base gives, with silver nitrate, a precipitate of the composition $(C_{13}H_{21}N)_2AgNO_3$. Symmetrical piperidines were prepared by the action of sodium on the alcoholic solutions of the corresponding pyridines by Ladenburg's process (Abstr., 1884, 1202). Symmetrical *trimethylpiperidine* or *copellidine*, $C_5NH_8Me_3$ [$Me_3 = 2:4:6$], boils between 145° and 147°. It is freely miscible with alcohol and ether, and forms crystalline salts, most of which dissolve freely in water. The platinochloride crystallises in orange plates and melts at 205°. Symmetrical *parpevoline* or *ethyl lupetidine*, C_5NH_8MeEt [$Me_2 : Et = 2:6:4$], boils at 165—167° under 725 mm. pressure. The *platinochloride*, $(C_9H_{19}N)_2,H_2PtCl_6$, and the *dichromate*, $(C_9H_{19}N)_2,H_2Cr_2O_7$, crystallise in prisms. Symmetrical *propyllupetidine*, $C_5NH_8Me_2Pr^a$, boils at 178—183° under 718·4 mm. pressure. The crystalline platinochloride melts at 197°. *Symmetrical isobutyllupetidine*, $C_5NH_8Me_2\cdot CH_2\cdot CHMe_2$, boils at 196—198° under a pressure of 720 mm. The hydrochloride and hydrobromide are crystalline, but the dichromate and platinochloride are not. *Symmetrical hexyllupetidine*, $C_5NH_8Me_2\cdot C_6H_{13}$, boils at 239—242° under 715 mm. pressure. The hydrochloride is crystalline.

The poisonous character of these piperidines increases with the molecular weight.

The minimal fatal dose for a frog is 0·02 gram of lupetidine, 0·01 gram of copellidine, 0·05 gram of parpevoline, or 0·004 gram of hexyllupetidine. In the case of poisoning with the piperidines, the red blood corpuscles are perforated, but the number and size of the holes diminishes as the molecular weight of the base increases.

W. C. W.

Conversion of Piperidine into δ-Amidovaleric Acid and into Oxypiperidine. By C. Schotten (*Ber.*, **21**, 2235—2254).—Benzoyl-δ-valeric acid is identical with benzoylhomopiperidinic acid (Abstr., 1885, 176), and can be obtained by acting on a mixture of 1 mol. of δ-amidovaleric acid and 1 mol. of benzoic chloride with an aqueous solution of 1 mol. of soda. The acid is decomposed into

benzoic acid and piperidine when distilled. The *barium* salt is anhydrous and readily soluble.

Benzoyl-δ-amidovaleric anhydride, $COPh\cdot C_5NH_8O$, obtained by boiling the acid with acetic anhydride, crystallises from alcohol in nacreous plates, melts at 112°, is sparingly soluble in ether, and insoluble in soda or sodium carbonate.

δ-Amidovaleric acid, $NH_2\cdot C_4H_8\cdot COOH$ (normal), prepared by heating the benzoyl-derivative with hydrochloric acid, crystallises in colourless, nacreous plates, is extremely soluble in water, less readily in dilute alcohol, almost insoluble in absolute alcohol, and insoluble in ether. It melts and decomposes at 157—158°, yielding water and oxypiperidine, and when heated with alkalis yields carbonic anhydride and butylamine. It forms salts both with acids and with bases, all of which are readily soluble in water. The *hydrochloride* crystallises in prisms. The *barium* salt is crystalline, and is not decomposed by carbonic anhydride.

Oxypiperidine, C_5H_9ON, formed as stated above, is a colourless, crystalline compound, melts at 39—40°, boils at 256°, and is extremely soluble in water, alcohol, ether, and dilute acids, but insoluble in concentrated potash or soda. It is precipitated from neutral or acid aqueous solutions by the usual reagents for alkaloïds, and when boiled with concentrated acids or alkalis is reconverted into δ-amidovaleric acid. Oxypiperidine is a tolerably strong poison, its action being very similar to that of strychnine. The *acetyl*-derivative, C_5H_8ONAc, is a colourless liquid, boils at 238°, and is readily soluble in water, alcohol, and ether.

Metanitrobenzoylpiperidine, $NO_2\cdot C_6H_4\cdot CO\cdot C_5NH_{10}$, is obtained by mixing 1 mol. of metanitrobenzoic chloride and 1 mol. of piperidine with a concentrated aqueous solution of 1 mol. of potash or soda. It crystallises from alcohol in dark yellow, pyramidal prisms, melts at 83—84°, and is readily soluble in alcohol and ethyl acetate, but rather sparingly in ether. It crystallises from hot water, in which it is only very sparingly soluble, in plates containing water (as much as 29·1 per cent.), the quantity of which seems to vary with the concentration and temperature of the solution; the crystals lose their water on exposure to the air. It dissolves in concentrated hydrochloric or fuming nitric acid, and is reprecipitated unchanged by water; when boiled with concentrated nitric acid, it is only attacked very slightly, a small quantity of butyric or valeric acid being formed. It yields small quantities of pyridine or bromopyridine when boiled with a glacial acetic acid solution of bromine.

Amidobenzoylpiperidine, prepared by reducing the preceding compound with ammonium sulphide, separates from hot water in long, colourless crystals, melts at 125°, and is soluble in alcohol, but almost insoluble in water. The *platinochloride*,

$$(NH_2\cdot C_6H_4\cdot CO\cdot C_5NH_{10})_2,H_2PtCl_6,$$

crystallises in shining needles, and is almost insoluble in water.

Metanitrobenzoyl-δ-amidovaleric acid, $NO_2\cdot C_6H_4\cdot CO\cdot NH\cdot C_5H_9O_2$, obtained by oxidising metanitrobenzoylpiperidine with potassium permanganate, crystallises from water in small, colourless, rhombic

tables, from alcohol in yellowish, shining plates, and from ethyl acetate in colourless prisms melting at 134—135°. It is almost insoluble in very dilute hydrochloric acid and in ether, rather easily in alcohol, but less readily in cold ethyl acetate. The *barium* salt, $(C_{12}H_{13}N_2O_5)_2Ba + 2\frac{1}{2}H_2O$, crystallises from water in rhombic and in hexagonal plates, which are readily soluble in water, but sparingly soluble in alcohol. The *cadmium* salt, $(C_{12}H_{13}N_2O_5)_2Cd + 7H_2O$, and the *zinc* salt are crystalline, but the *silver* salt is amorphous. The acid yields metanitrobenzoic acid and δ-amidovaleric acid when boiled with concentrated hydrochloric acid. The *anhydride*, $NO_2{\cdot}C_6H_4{\cdot}CO{\cdot}N{:}C_5H_8O$, obtained by boiling the acid with acetic anhydride, crystallises from dilute alcohol in shining, yellowish plates melting at 114°.

Parabromobenzoic chloride crystallises in long, colourless prisms, melts at 42°, and boils at 174° (102 mm. pressure.)

Parabromobenzamide crystallises from hot water in colourless needles, melts at 190°, and is rather easily soluble in alcohol, but sparingly soluble in ether.

Parabromobenzoylpiperidine, $C_6H_4Br{\cdot}CO{\cdot}C_5NH_{10}$, prepared from parabromobenzoic chloride and piperidine, crystallises from alcohol in large, clouded, rectangular plates, melts at 95°, and is readily soluble in alcohol, but only sparingly in ether or water.

Parabromobenzoyl-δ-amidovaleric acid, $C_6H_4Br{\cdot}CO{\cdot}NH{\cdot}C_5H_9O_2$, prepared by oxidising the preceding compound with potassium permanganate, crystallises from 90 per cent. hot alcohol in needles, melts at 180—181°, and is sparingly soluble in water and ether. It yields a crystalline anhydride when boiled with acetic anhydride. The alkaline salts are readily soluble in water. The *barium* salt, $(C_{12}H_{13}BrNO_3)_2Ba$, crystallises from hot water, in which it is readily soluble, in nacreous plates.

Orthobromobenzoic chloride is a colourless liquid boiling at 158° (49 mm. pressure). *Orthobromobenzamide* crystallises from hot water in needles, melts at 155—156°, and is sparingly soluble in ether.

Orthobromobenzoylpiperidine is a thick, yellowish oil, and behaves towards solvents like the corresponding para-compound.

Orthobromobenzoyl-δ-amidovaleric acid, obtained by oxidising the preceding compound with potassium permanganate, is a crystalline compound which melts at 110—111°, and is readily soluble in alcohol, but only sparingly in ether or water. It yields a crystalline anhydride when boiled with acetic anhydride. The alkaline salts and those of the alkaline earths are readily soluble in water, but those of the heavy metals only sparingly. The *silver* salt, $C_{12}H_{13}BrNO_3Ag$, is crystalline.

Salicylpiperidine, $OH{\cdot}C_6H_4{\cdot}CO{\cdot}C_5NH_{10}$, is formed when methyl salicylate is heated with anhydrous piperidine. It crystallises from alcohol in thin, yellowish plates, melts at 142°, is sparingly soluble in ether, and almost insoluble in cold water and dilute acids, but readily soluble in hot soda.

Parahydroxybenzoylpiperidine, $OH{\cdot}C_6H_4{\cdot}CO{\cdot}C_5NH_{10}$, is obtained when piperidine parahydroxybenzoate is treated with phosphorus chloride in the cold. It crystallises from dilute alcohol in thick

prisms, melts at 210°, and behaves towards solvents like salicylpiperidine. F. S. K.

Tertiary Phenylpiperidine. By E. Lellmann and W. Geller (*Ber.*, **21**, 2279—2281 (compare Lellmann, Abstr., 1887, 604, and Lellmann and Geller, this vol., p. 970).—The halogen-derivatives of benzene react with phenylpiperidine, yielding tertiary phenylpiperidine. Tertiary phenylpiperidine is obtained when bromobenzene is heated with piperidine (3 mols.) at 250—260° for 24 hours, the product filtered from piperidine hydrobromide and fractioned; also when an alcoholic solution of amidophenylpiperidine is treated with ethyl nitrite and concentrated sulphuric acid. It is a bright yellow liquid of disagreeable odour, boils at 245—250°, and is a strong base.

The *platinochloride*, $(C_5NH_{10}Ph)_2,H_2PtCl_6 + 2H_2O$, crystallises in needles or plates, and is partially decomposed when heated in solution. Mercuric chloride produces an oily precipitate in a hydrochloric acid solution, and potassium dichromate precipitates small, shining, crystalline plates. F. S. K.

Derivatives of Tertiary Phenylpiperidine. By E. Lellmann and W. Geller (*Ber.*, **21**, 2281—2287).—*Orthonitrophenylpiperidine*, $NO_2\cdot C_6H_4\cdot C_5NH_{10}$, prepared by heating orthonitrobromobenzene with piperidine, crystallises from alcohol in large, ruby-red prisms, melts at 81°, and is readily soluble in alcohol, ether, benzene, and chloroform. The *hydrochloride*, $NO_2\cdot C_6H_4\cdot C_5NH_{10},HCl$, is a yellow, crystalline compound, and is decomposed by water. The *platinochloride*, $(NO_2\cdot C_6H_4\cdot C_5NH_{10})_2,H_2PtCl_6$, crystallises in shining leaves.

Paranitrophenylpiperidine, $NO_2\cdot C_6H_4\cdot C_5NH_{10}$, prepared from parachloronitrobenzene and piperidine, crystallises from alcohol in large, yellow plates, which have a slight blue colour by reflected light, and melt at 105·5°. It is readily soluble in alcohol, chloroform, benzene, and ether. The *hydrochloride*, $NO_2\cdot C_6H_4\cdot C_5NH_{10},HCl$, is a bright yellow, crystalline compound, and is decomposed by water. The *platinochloride*, $(NO_2\cdot C_6H_4\cdot C_5NH_{10})_2,H_2PtCl_6$, forms small, yellow crystals.

Orthoparadinitrophenylpiperidine, $C_6H_3(NO_2)_2\cdot C_5NH_{10}$, prepared by gradually adding piperidine to finely-divided dinitrochlorobenzene and heating the mixture, crystallises from alcohol in orange-yellow needles with a slight blue colour by reflected light. It melts at 92°, and is readily soluble in ether, benzene, and chloroform.

Parachlororthonitrophenylpiperidine, $NO_2\cdot C_6H_3Cl\cdot C_5NH_{10}$, the reaction-product of nitroparadichlorobenzene and piperidine, is a bright red, crystalline compound, melts at 51°, and is readily soluble in alcohol, ether, benzene, and chloroform.

Chloronitroamidophenylpiperidine, $NO_2\cdot C_6H_2Cl(NH_2)\cdot C_5NH_{10}$, is obtained when a concentrated hydrochloric acid solution of stannous chloride is gradually added to a boiling, concentrated hydrochloric acid solution of dinitrophenylpiperidine. It is a crystalline substance, melts at 111·5°, and is soluble in alcohol, benzene, ether and chloroform.

Paramidophenylpiperidine hydrochloride, $NH_2\cdot C_6H_4\cdot C_5NH_{10},2HCl + H_2O$, prepared by treating paranitrophenylpiperidine with a hot,

concentrated hydrochloric acid solution of stannous chloride, is a yellow compound, and is readily soluble in water and alcohol. Ferric chloride produces a red coloration in a neutral or slightly acid solution of the salt. The free *base* is a colourless, crystalline compound, melts at 40°, and is readily soluble in all ordinary solvents except water; it yields tertiary phenylpiperidine when treated with ethyl nitrite and concentrated sulphuric acid in alcoholic solution. The *hydrochloride* of the acetyl-derivative, $NH{\cdot}COMe{\cdot}C_6H_4{\cdot}C_5NH_{10},HCl$, is a colourless, crystalline compound, and readily soluble in water; a subcutaneous dose of 0·3 gram proved fatal to a rabbit within a few minutes. The *acetyl*-derivative, $NH{\cdot}COMe{\cdot}C_6H_4{\cdot}C_5NH_{10}$, crystallises from alcohol in colourless, nacreous plates, melts at 151°, and is readily soluble in alcohol and chloroform, moderately soluble in ether and benzene, but very sparingly soluble in water. F. S. K.

Formation of Colouring Matters from Paramidophenylpiperidine. By E. Lellmann and W. Geller (*Ber.*, **21**, 2287—2288).—Indamines with a bluish or greyish shade are obtained when a cold, neutral solution of a mixture of paramidophenylpiperidine hydrochloride and an equivalent quantity of the hydrochloride of a primary, secondary, or tertiary aromatic amine is oxidised with the theoretical quantity of potassium dichromate, and the colouring matter precipitated with zinc chloride. If oxidation takes place in presence of a metadiamine, for example, metaphenylene- or metatoluylene-diamine, intensely blue-coloured substances are produced; and, on adding zinc chloride, these are precipitated in the form of a brown powder. These colouring matters belong to the same group as toluylene-blue, as is shown by the change of colour which occurs when an acid solution is boiled; for example, the toluylene-derivative gives a red, the phenylene colour a more violet dye, both of which are precipitated from concentrated solutions by zinc or sodium chloride. These indamines can be converted into safranines in the usual manner. A safranine which dyes blue is obtained when a mixture of amidophenylpiperidine hydrochloride (1 mol.) and aniline hydrochloride (2 mols.) is oxidised in cold, dilute acetic acid solution.

A blue indophenol separates when a dilute alkaline solution of paramidophenylpiperidine (1 mol.) and phenol (1 mol.) is oxidised with the theoretical quantity of potassium ferricyanide, or when a dilute acetic acid solution of amidophenylpiperidine hydrochloride and phenol is oxidised with ferric chloride. The naphthols yield compounds of a more distinctly violet colour, amongst which that derived from β-naphthol is distinguished by its reddish shade and its solubility in soda.

An intensely blue substance is produced when a hydrochloric acid solution of amidophenylpiperidine is treated with hydrogen sulphide and ferric chloride, and the product precipitated with zinc chloride and sodium chloride. It dyes a somewhat greenish colour in comparison to methylene-blue. F. S. K.

Orthonitroparahydroxyquinoline and Orthamidoparahydroxyquinoline. By J. Altschul (*Ber.*, **21**, 2254—2255).—The

nitrohydroxyquinoline described by Matheus (this vol., pp. 851, 965) is identical with the compound obtained by the author (this vol., p. 67) from parahydroxyquinolinecarboxylic acid, and also with the compound obtained by Skraup (Abstr., 1883, 92) from parahydroxyquinoline. The *acetyl*-derivative of amidoparahydroxyquinoline loses water when heated at 235°, and yields a crystalline ethenyl-derivative.

F. S. K.

γ-Hydroxyquinaldine. By M. Conrad and L. Limpach (*Ber.*, **21**, 1965—1984).—*Methyl phenylamidocrotonate*, $C_{11}H_{13}NO_2$, is obtained when equivalent quantities of methyl acetoacetate and aniline are mixed together. It crystallises from methyl alcohol in large, compact prisms, melts at 51°, and, when heated quickly to 240—250°, yields about 40 per cent. of hydroxyquinaldine.

Ethyl quinaldylcarboxylate, $C_{10}NH_8{\cdot}COOEt$, is formed when ethyl chloroformate is added to well-dried sodiohydroxyquinaldine suspended in benzene. It crystallises in white, shining prisms, melts at 48°, and is readily soluble in ether, alcohol, and hot benzene, but insoluble in water. It is decomposed into hydroxyquinaldine, carbonic anhydride, and alcohol when heated with water, or more quickly by acids or alkalis. The *platinochloride*, $(C_{13}H_{13}NO_3)_2,H_2PtCl_6 + 2H_2O$, is a yellow, crystalline compound, and when heated to 100° melts and then solidifies; on further heating, it melts again at 130° with decomposition and evolution of gas.

Quinaldylbenzoate, $C_{10}H_8NO{\cdot}COPh$, prepared by treating sodiohydroxyquinaldine with benzoic chloride, is very readily soluble in ether, alcohol, and benzene, and crystallises in large, well-defined prisms melting at 129°. It yields benzoic acid and hydroxyquinaldine when warmed with water, or more readily with acids or alkalis. The *platinochloride* is a yellow, crystalline compound, and melts at 180—190° with decomposition.

Sodium hydroxyquinaldineazobenzenesulphonate is formed when γ-hydroxyquinaldine is treated with sodium paradiazobenzenesulphonate in alkaline solution. It crystallises from hot water in orange-red needles, and when warmed with stannous chloride and hydrochloric acid yields sulphanilic acid and amido-γ-hydroxyquinaldine hydrochloride. Amido-γ-hydroxyquinaldine (Abstr., 1887, 680) separates in white, shining prisms when ammonia is added to a solution of this hydrochloride. The constitution of hydroxyquinaldineazobenzenesulphonic acid is therefore $OH{\cdot}C_9NH_4Me{\cdot}N_2{\cdot}C_6H_4{\cdot}SO_3H$ [$N_2 = 3'$].

Thioquinaldine hydrochloride, $C_{10}H_9NS,HCl$, crystallises in white needles, melts at 205—210° with decomposition, and is readily soluble in water.

γ-Hydroxyquinaldinealdehyde,

$$OH{\cdot}C_9NH_4Me{\cdot}CHO\ [Me : CHO : OH = 2' : 3' : 4'],$$

is obtained when chloroform is added gradually to a hot alkaline solution of hydroxyquinaldine and the mixture heated for five to six hours. It crystallises from boiling alcohol in bright yellow, transparent plates, which melt at 273° with decomposition; it is readily soluble in dilute alkalis and concentrated hydrochloric acid, but only sparingly in ether, benzene, or water. The *hydrochloride*,

$C_{11}H_9NO_2,HCl$, dissociates when heated with water. The *platinochloride* $(C_{11}H_9NO_2)_2,H_2PtCl_6$, is a yellow, crystalline compound; it melts at 215—220° with decomposition, and is soluble in hot water. The hydrochloride, $C_{17}H_{15}N_3O,HCl$, of the *hydrazide* crystallises in slender, yellow needles, and is relatively easily soluble in alcohol, but only sparingly soluble in hot water. A condensation product, $C_{31}H_{25}N_3O_3$, is formed in the preparation of hydroxyquinaldinealdehyde. It is soluble in hot benzene and boiling ethyl acetate, and melts at 192°. When heated with water, the larger portion dissolves and γ-hydroxyquinaldine separates from the filtered and concentrated solution.

γ-Hydroxyquinaldine-β-carboxylic acid, $OH{\cdot}C_9NH_4Me{\cdot}COOH$ [= 4′ : 2′ : 3′], is readily obtained by oxidising the aldehyde with alkaline potassium permanganate. It is almost insoluble in ether, benzene, and boiling water, but readily soluble in alcohol, and melts at 245° with evolution of carbonic anhydride and formation of γ-hydroxyquinaldine. An aqueous solution of the ammonium salt gives a red coloration with ferric chloride, and yields precipitates with lead acetate, copper sulphate, mercuric nitrate, and silver nitrate. The *zinc* salt crystallises in needles. The *magnesium* salt, $(C_{11}H_8NO_3)_2Mg$, forms white, opaque crystals.

Trimethyl-γ-hydroxyquinaldinealdehyde, $C_6HMe_3\langle \begin{matrix} N{\cdot}CMe \\ | \\ C(OH) \end{matrix} \rangle C{\cdot}CHO$, is formed when alcoholic chloroform is added gradually to a hot alkaline solution of trimethylhydroxyquinaldine and the mixture heated for several hours. The reaction-product is dissolved in dilute hydrochloric acid and treated with phenylhydrazine hydrochloride, when the hydrochloride of the *hydrazide*, $C_{20}H_{22}N_3O,HCl$, separates as a yellow, crystalline salt, which is sparingly soluble in water and alcohol.

γ-Hydroxyquinaldinesulphonic acid, $C_{10}NH_8O{\cdot}SO_3H$, prepared by heating γ-hydroxyquinaldine at 110—115° with fuming sulphuric acid, crystallises from hot water in long, shining prisms, which contain 2 mols. of water, and are readily soluble in hot alcohol. The anhydrous substance melts at 283°.

The *barium* salt, $(C_{10}NH_8O{\cdot}SO_3)_2Ba + 4H_2O$, crystallises from boiling water in large, shining prisms. A dilute, neutral, aqueous solution of the ammonium salt gives precipitates with barium chloride, calcium chloride, lead acetate, and ferric chloride.

Diazohydroxyquinaldineanhydride, $C_9NH_4Me\langle \begin{matrix} N \\ O \end{matrix} \rangle N$ [Me : N : O = 2′ : 3′ : 4′], is formed when sodium nitrite is added to a dilute sulphuric acid solution of amidohydroxyquinaldine. It crystallises from ether and benzene in shining, yellow needles, and melts at 129—131° with sudden decomposition and evolution of gas. The *hydrochloride*, $C_{10}H_7N_3O,HCl$, separates as a reddish, crystalline precipitate when hydrochloric acid is added to an ethereal solution of the preceding compound. The hydrochloride of amidohydroxyquinaldine is obtained when a concentrated hydrochloric acid solution of diazohydroxyquinaldineanhydride is reduced. Acetanthranilic acid is formed when amidohydroxyquinaldine is oxidised with chromic acid.

Amidoquinaldine, $C_9NH_5Me{\cdot}NH_2$ [= 2′ : 3′], prepared by heating amidohydroxyquinaldine at 180—210° with an acetic acid solution of hydrogen iodide, is a colourless oil which boils at 270° and has a strongly alkaline reaction. The *picrate* is a sparingly soluble, yellow, crystalline compound. The *hydrochloride* is white, crystalline, and very hygroscopic. The *platinochloride*, $(C_{10}H_{10}N_2)_2,H_2PtCl_6$, is yellow, crystalline, and soluble in hot water.

Nitrochloroquinaldine, $C_9NH_4ClMe{\cdot}NO_2$ [Me : NO_2 : Cl = 2′ : 3′ : 4′], is obtained when nitrohydroxyquinaldine is heated with a mixture of phosphorus pentachloride and phosphorus oxychloride. It separates from ether or hot benzene in yellowish crystals, is readily soluble in boiling alcohol, sparingly soluble in water, and melts at 93—94°. It is reconverted into nitrohydroxyquinaldine when gently heated with water.

Nitramidoquinaldine, $NO_2{\cdot}C_9NH_4Me{\cdot}NH_2$ [Me : NO_2 : NH_2 = 2′ : 3′ : 4′], is prepared by saturating a benzene solution of nitrochloroquinaldine with ammonia, adding a small quantity of concentrated alcoholic ammonia and heating the mixture at 180—200°. It crystallises from alcohol in slender, bright yellow needles melting at 201°.

Diamidoquinaldine hydrochloride, $C_{11}H_{11}N_3,HCl$, is obtained in colourless crystals when the preceding compound is reduced.

Trichloroquinaldine, $C_{10}H_6NCl_3$, obtained in the preparation of chloroquinaldine (*loc. cit.*), crystallises from alcohol in white needles, melts at 102°, and is soluble in ether. F. S. K.

Syntheses with Ethyl Acetoacetate. By L. Knorr (*Annalen*, **245**, 357—382; compare Abstr., 1887, 159, 275, and 601).—It was previously mentioned (*Annalen*, **236**, 112) that besides ethyl acetoacetate, methylacetoacetic and benzoylacetic acids form anilides which can be condensed to quinolines.

Methylacetoacetic anilide, $COMe{\cdot}CHMe{\cdot}CO{\cdot}NHPh$, is prepared by heating carefully purified ethyl methylacetoacetate and aniline at 150—160°. It crystallises from hot water in prisms, melts at 138—140°, is soluble in ether, benzene, and chloroform, sparingly soluble in hot water.

Dimethylcarbostyril, [Me_2 = 3′ : 4′], is obtained on keeping the above anilide dissolved in cold concentrated sulphuric acid for a long time, and may be purified by means of the hydrochloride; it melts at 262°. The *hydrochloride* crystallises from the strong acid solution in long, lustrous needles which give off hydrogen chloride when kept in a desiccator over potash, and are completely decomposed by water. The *sulphate* is decomposed by alcohol. Dimethylcarbostyril is also a very feeble acid. The *sulphonic acid* is prepared by heating the compound with strong sulphuric acid at 200°. The *barium sulphonate* was prepared.

Chlorodimethylquinoline, $C_{11}H_{10}NCl$ [Cl : Me_2 = 2′ : 3′ : 4′], is formed when 3 grams of dimethylcarbostyril and 5 grams of phosphorus pentachloride are heated at 180°. It melts at 131°, distils with steam, and is a feeble base. The *platinochloride*, $(C_{11}H_{10}NCl)_2,H_2PtCl_6 + 4H_2O$, forms orange-coloured crystals. When the free base is heated with water at 200°, it is converted into dimethylcarbostyril.

Dimethylquinoline, [$Me_2 = 3' : 4'$], is obtained by distilling dimethylcarbostyril over heated zinc-dust. The product is dissolved in sulphuric acid, extracted with ether, the solution of the sulphate treated with soda, and steam distilled. It melts at 65°, and boils at 290° under 737 mm. pressure. The *hydrochloride* crystallises in prisms; the *platinochloride* (with 2 mols. H_2O) forms flat needles; the *picrate* crystallises in red, lustrous needles, melting at about 205°, the *aurochloride*, *argentonitrate*, and *methiodide* melting at 177°, 180°, and 190—191° respectively are also described.

Paradimethylcarbostyril, [Me : Me = 4' : 3], (Abstr., 1884, 1198), crystallises in flat prisms of a satiny lustre, and melts at 249—250°; it dissolves very sparingly in ether, chloroform, benzene, and light petroleum, but is soluble in alcohol. It is slightly basic as well as acid. The *hydrochloride* and *nitrate* crystallise in hair-like needles; the *sulphate* forms short prisms; all three salts are decomposed by water. The compound behaves towards bromine-water, phosphorus pentachloride, sodium ethoxide, and methyl iodide just like oxylepidine.

Paradimethylquinoline, $C_{11}H_{11}N$, prepared by distilling 4' : 3-dimethylcarbostyril with zinc-dust, boils at 280° (under 754 mm. pressure), dissolves readily in alcohol and ether, hardly at all in water, has a burning taste, and a penetrating odour like that of quinoline. The *platinochloride* (with 2 mols. H_2O) crystallises in bent needles which decompose at 231°; the *picrate* separates in branched needles which decompose at about 230°; the *aurochloride* forms bright yellow needles and decomposes at about 190°; the *chromate*, *sulphate*, and *argentonitrate* are described.

Orthodimethylcarbostyril, [$Me_2 = 4' : 1$] (*loc. cit.*), crystallises in needles sparingly soluble in water, insoluble in ammonia and alkaline carbonates. The *sodium salt* forms thin plates. The *hydrochloride* and *nitrate* crystallise in needles, and are decomposed by water. The *platinochloride* (with 2 mols. H_2O) was prepared.

Orthodimethylquinoline, [$Me_2 = 4' : 1$], is a bright yellow oil, readily soluble in alcohol and ether; it has an odour like that of quinoline. The *platinochloride* melts at about 220° with decomposition; the *aurochloride* forms lustrous lozenge-shaped crystals melting at 181°; the *chromate*, *picrate*, and *argentonitrate* are also described.

Metadimethylcarbostyril, $C_{11}H_{11}NO$, melts at 220° and dissolves very sparingly in hot water. The *hydrochloride* and *nitrate* crystallise in hair-like needles, and are decomposed by water; the *platinochloride* forms yellow needles which decompose at 233—234°.

Metadimethylquinoline, $C_{11}H_{11}N$, boils at about 283° (under 750 mm. pressure). The *platinochloride* (with 2 mols. H_2O) crystallises from hot hydrochloric acid in well-formed, short, reddish-brown prisms which decompose at 227°.

Methyl β-phenylamidophenylacrylate, $C_{18}H_{15}NO_2$, separates in prisms melting at 92—93° when a mixture of aniline and methyl benzoylacetate is kept for some weeks at the ordinary temperature.

β-Phenylamidophenylacrylic anilide, NHPh·CPh:CH·CO·NHPh, is formed together with benzoylacetic anilide when the mixture of aniline and methyl benzoylacetate is heated for a long time at 150°. It crystallises in needles, melts at 133°, is almost insoluble in water,

dilute acids, and alkalis, soluble in ether, hot benzene, alcohol, and chloroform. Boiling dilute acids decompose it into aniline and benzoylacetic anilide. When distilled, it decomposes into aniline, diphenylcarbamide, and a compound of the formula $C_{15}H_{11}NO$, probably phenylhydroxyquinoline.

Benzoylacetic anilide, $COPh{\cdot}CH_2{\cdot}CO{\cdot}NHPh$, crystallises in plates, melts at 107—109°, dissolves readily in alcohol, chloroform, and hot benzene, sparingly in hot water and dilute hydrochloric acid. It is dissolved by alkalis, and precipitated by carbonic anhydride. The alcoholic solution gives a blue-violet coloration with ferric chloride. The *isonitroso-derivative*, $C_{15}H_{12}N_2O_3$, crystallises in needles melting at about 190°. When the solution of the anilide in methyl alcohol is saturated with dry hydrogen chloride, the *hydrochloride*, $C_{15}H_{13}NO_2,HCl$, separates after some hours in lustrous prisms; it melts at 99°, and is extremely soluble in methyl alcohol.

Phenylhydroxyquinoline, $C_{15}H_{11}NO$ (Just, Abstr., 1886, 811), is obtained by heating the above anilide with strong sulphuric acid in a water-bath; a too prolonged heating is to be avoided; no action takes place in the cold. It melts at 254°, dissolves in hot alcohol, readily in alkalis. The alkaline salts are decomposed by carbonic anhydride. The *nitrate* crystallises in hair-like needles; the *hydrochloride* forms splendid needles; the *platinochloride* crystallises in small, lozenge-shaped crystals. When phenylhydroxyquinoline is distilled with zinc-dust, phenylquinoline (Doebner and v. Miller, Abstr., 1883, 1149; and Königs and Nef, Abstr., 1886, 1045), (m. p. 86°), is formed. The *aurochloride*, $(C_{15}H_{11}N)_2,H_2AuCl_5$, crystallises in needles melting at 204°; another *aurochloride*, $C_{15}H_{11}N,HAuCl_4$, melting at 160°, was obtained. N. H. M.

α-Amidolepidine. By C. Klotz (*Annalen*, **245**, 382—384).—*α-Amidolepidine*, $C_{10}H_{10}N_2$, obtained by the action of alcoholic ammonia on α-chlorolepidine at 200—230°, is a crystalline powder, melts at 131—132°, dissolves readily in ether, chloroform, alcohol, glacial acetic acid, and hot benzene. The *sulphate* crystallises in needles melting at 155°; the *picrate* forms sulphur-coloured needles which melt at about 250°; the *platinochloride* crystallises in orange-coloured needles and melts at 230°. N. H. M.

Lepidine-compounds. By B. Heymann and W. Koenigs (*Ber.*, **21**, 2167—2172; compare this vol., p. 852).—*Orthohydroxybenzyllepidine*, $C_9NH_6{\cdot}CH_2{\cdot}CH_2{\cdot}C_6H_4{\cdot}OH$, is obtained by reducing orthohydroxybenzylidenelepidine (*loc. cit.*) with hydriodic acid and amorphous phosphorus in glacial acetic acid solution. It crystallises from hot aqueous alcohol in colourless needles, melts at 180—181°, and is readily soluble in cold absolute alcohol, but only sparingly in dilute alcohol. It dissolves very sparingly in cold, but readily in hot, dilute soda, and a crystalline sodium salt separates from the solution on cooling. The *hydrochloride* is sparingly soluble in cold, very dilute hydrochloric acid, and separates from the hot solution in an oily condition. The *hydrobromide* behaves similarly. Salicylic acid is obtained when the base is fused with alkali and a small

quantity of water, but no hydroxyquinoline-derivative is formed, this is also the case when the para- and meta-compound are treated in the same way.

Meta-amidobenzylidenelepidine, $C_9NH_6{\cdot}CH{:}CH{\cdot}C_6H_4{\cdot}NH_2$, prepared by reducing metanitrobenzylidenelepidine (*loc. cit.*) with stannous chloride and hydrochloric acid in alcoholic solution, crystallises from absolute alcohol in shining, yellow needles or leaves, melts at 141°, and is readily soluble in dilute hydrochloric acid, dilute sulphuric acid, and hot alcohol.

Metahydroxybenzylidenelepidine, $C_9NH_6{\cdot}CH{:}CH{\cdot}C_6H_4{\cdot}OH$, is obtained when a dilute solution of the preceding compound is mixed with sodium nitrite and boiled. It separates from hot, absolute alcohol in brownish-yellow, lance-shaped crystals melting at 254—255°, and is sparingly soluble in soda and boiling alcohol. The *hydrochloride* is sparingly soluble in cold, more readily in hot water, and crystallises from hot, absolute alcohol, in which it is readily soluble, in brownish needles.

Metahydroxybenzyllepidine, $C_9NH_6{\cdot}CH_2{\cdot}CH_2{\cdot}C_6H_4{\cdot}OH$, prepared by reducing the preceding compound with hydriodic acid and amorphous phosphorus, crystallises from hot alcohol, in which it is sparingly soluble, in prisms melting at 209°. It is rather soluble in cold, dilute soda, and is precipitated from the solution by hydrochloric acid in the form of a sparingly soluble, oily *hydrochloride*. When fused with an alkali, it yields considerable quantities of metahydroxybenzoic acid. Parahydroxybenzyllepidine yields parahydroxybenzoic acid when treated similarly, and a small quantity of paracresol appears also to be formed.

Benzylquinaldine and benzyllepidine, when carefully heated above 350°, distil with only slight decomposition. *Benzyllepidine nitrate* separates from a hot, very dilute nitric acid solution of the base in sparingly soluble crystals. Cinchonic acid is formed in considerable quantity when benzylidenelepidine is boiled for several days with an acetic acid solution of chromic acid.

The melting point of most of the compounds described in the previous paper (*loc. cit.*) are given 4° too high. F. S. K.

Isoquinoline and its Derivatives. By M. Le Blanc (*Ber.*, **21**, 2299—2301).—Isoquinoline can be prepared by heating a mixture of homoorthophthalimide and zinc-dust to a dull red heat in a stream of hydrogen. The yield is about 40 per cent. It is also formed when methyl homoorthophthalimide is distilled. *γ-Methyl isoquinoline*, $C_6H_4{<}\begin{matrix}CMe{:}CH\\CH{=}N\end{matrix}{>}$, is obtained by distilling di- or tri-methyl homoorthophthalimide with zinc-dust; it boils at 256°. The *picrate* crystallises in slender needles melting at 194—195°. The platinochloride is a brownish-red crystalline compound melting at 253·5°. F. S. K.

New Vegetable Base. By A. Kossel (*Ber.*, **21**, 2164—2167).—A new base, $C_7H_8N_4O_2$, which the author names *thiophylline*, exists in small quantities in tea extract, and occurs together with caffeïne. It crystallises with 1 mol. of water, which it loses at 110°, is considerably

more soluble in alcohol and water than theobromine, and is extremely soluble in very dilute ammonia. It melts at 264°, forms crystalline salts with hydrochloric acid, nitric acid, platinum tetrachloride, auric chloride, and mercuric chloride, and combines with soda to form a readily soluble compound. When thiophylline is evaporated with chlorine-water, a scarlet residue is obtained, which changes to violet when treated with ammonia.

The *silver*-derivative, $C_7H_7N_4O_2Ag$, obtained in the form of an amorphous precipitate by adding silver nitrate to an aqueous solution of the base, crystallises from hot ammonia, and is readily soluble in nitric acid. The *methyl*-derivative, $C_8H_{10}N_4O_2$, prepared by heating the silver-derivative with methyl iodide and methyl alcohol, melts at 229°, and is identical with caffeïne. Thiophylline is, therefore, dimethylxanthine. F. S. K.

Morphine. By O. Hesse (*Pharm. J. Trans.*, **18**, 801).—The author questions the accuracy of the formula $8C_{17}H_{19}NO_3 + 9H_2O$, assigned to crystalline morphine by Dott (this vol., p. 506), as his own experiments have shown exactly 1 mol. H_2O for each molecule of morphine. The melting point, according to his observations, is 230° (with decomposition), instead of 120°, as stated in the books. In the delicate test for morphine proposed by Armitage (this vol., p. 1137) the reaction is not the result of reduction of the ferric chloride, but of the formation of pseudomorphine and ferrocyanic acid, thus:—

$$8(C_{17}H_{19}NO_3,HCl) + 8(K_3FeCy_6) = 4(C_{34}H_{36}N_2O_6,2HCl) + 6K_4FeCy_6 + 2H_4FeCy_6.$$

Pseudomorphine is actually deposited if the solution is not too dilute, and this substance being somewhat soluble in water, is the cause of the blue coloration in the presence of ferric chloride. R. R.

Narcotine. By W. Roser (*Annalen*, **245**, 311—326).—*Iodotarconine methiodide*, $C_{11}H_8INO_3,MeI$, is obtained together with tarconine methiodide when narcotine dissolved in 20 parts of 80 per cent. alcohol, and some hydrochloric acid is treated with the corresponding amount of iodine in three portions, and boiled in a reflux apparatus for 10 hours. The periodide which separates is dissolved in water, and treated with hydrogen sulphide. The liquid is warmed and quickly filtered, by which a separation of the sparingly soluble iodotarconine methiodide from the more readily soluble tarconine methiodide is effected. It is almost insoluble in boiling alcohol, and crystallises from hot water in three forms. When heated with a solution of iodine, the *periodide*, $C_{11}H_8INO_2,MeI,I_2$, is obtained. This crystallises in sparingly soluble, dark, lustrous needles melting at 171°.

Iodotarconine methochloride, $C_{11}H_8INO_3,MeCl + H_2O$, prepared by treating an aqueous solution of the iodide with silver chloride, crystallises from alcohol in yellowish needles. The *platinochloride*, $(C_{12}H_{11}INO_3)_2,PtCl_4$, crystallises is short, lustrous prisms; the *aurochloride* forms slender, yellow needles; the *mercurichloride* crystallises in long needles.

Iodotarconine, $C_{11}H_8INO_3 + H_2O$, is formed when iodotarconine methochloride is heated gradually up to 180°. It crystallises from water in yellowish-red needles. The anhydrous compound is dark-red. The *hydrochloride*, $C_{11}H_8INO_3,HCl + 2H_2O$, crystallises in yellow needles of silky lustre.

Tarconine methiodide, $C_{11}H_9NO_3,MeI$, prepared as described above, crystallises in groups of long, yellow needles; it combines with iodine with formation of the *periodide*, $C_{11}H_9NO_3,MeI,I_2$; it crystallises from alcohol in long, red-brown needles melting at 160° (compare Jörgensen, *J. pr. Chem.* [2], **2**, 433). When 1 gram of methyltarconine chloride is heated with 7 grams of strong hydrochloric acid for four hours at 140—150°, a compound, $C_{11}H_9NO_3,HCl + 1\frac{1}{2}H_2O$, probably tarconine hydrochloride (Wright, Trans., 1877, 535), and methyl chloride are formed.

Methyltarconic acid, $C_{11}H_{11}NO_2$, is formed together with formaldehyde when the solution of the ammonium base obtained by the action of silver oxide on tarconine methochloride is boiled; it separates in small, lustrous crystals. It is insoluble in alcohol, almost insoluble in water, readily soluble in mineral acids, and in alkalis (not ammonia). The *hydrochloride* crystallises in slender, lustrous needles, very readily soluble in water, sparingly in alcohol. The *sulphate*, $(C_{11}H_{11}NO_3)_2,H_2SO_4 + 6H_2O$, and the *acid sulphate*, $C_{11}H_{11}NO_3,H_2SO_4 + 3H_2O$, crystallise in small, yellow crystals and white prisms.

Bromotarconine methobromide, $C_{11}H_8BrNO_3,MeBr + H_2O$, is prepared by adding bromine-water to an aqueous solution of tarconine methochloride, and decomposing the perbromide which separates with hydrogen sulphide. It first separates in a labile form, in long sulphur-coloured needles; these gradually change to flat prismatic crystals.

The *perbromide*, $C_{11}H_8BrNO_3,MeBr,Br_2$, crystallises in lustrous, gold-coloured plates, melts at 165°, and is converted by boiling with alcohol into the bromide just described.

Bromotarconine methochloride, prepared by digesting the bromide with silver chloride, crystallises in slender needles, very readily soluble in alcohol, less in water. The *platinochloride* is yellowish-red; the *aurochloride* forms slender yellow needles.

Methylbromotarconic acid, $C_{11}H_{10}BrNO_3$ (v. Gerichten, Abstr., 1883, 869), is formed as barium salt when bromotarconine methobromide is treated with silver oxide, and the whole boiled with baryta. It melts at 233°. N. H. M.

Papaverine. By G. Goldschmiedt (*Monatsh.*, **9**, 327—348; compare Abstr., 1886, 83, 478; 1887, 163; this vol., p. 302)—Crystalline papaverine benzyl chloride (30 grams) is oxidised with 2 per cent. potassium permanganate (100 grams) at 40—45°, and the solution filtered. The filtrate contains veratric acid (7½ grams) and small quantities of benzoic and oxalic acids. The residue is either extracted consecutively with boiling water and alcohol, or suspended in water, and the oxide of manganese dissolved by passing a stream of sulphurous anhydride. The mixture of organic compounds is warmed with dilute hydrochloric acid and the solution filtered; on

adding ammonia to the filtrate, papaveraldine is precipitated. The residue contains a compound melting at 153—154° (see below), benzylhemipinisoimide, and a very small quantity of a compound melting above 240° which was not obtained in the pure state.

The substance melting at 153—154° is *benzylpapaveraldine-ammonium hydroxide*, $C_7H_7 \cdot C_{20}H_{19}NO_5 \cdot OH$, or *oxide* $(C_{20}H_{19}NO_5 \cdot C_7H_7)_2O$. It crystallises from boiling water or alcohol in small, slender, colourless needles, and is readily soluble in cold glacial acetic acid, acetone, chloroform, and benzene, but rather sparingly soluble in ether. It dissolves in boiling concentrated hydrochloric acid, yielding a yellow solution from which yellow needles of papaveraldine hydrochloride separate on cooling; it also dissolves in cold sulphuric acid, forming a colourless solution which shows the characteristic colour reaction of papaveraldine when heated. The *hydrochloride* crystallises in slender, yellow needles, but it quickly decomposes when dry and loses its acid.

Benzylhemipinisoimide, $C_{17}H_{15}NO_4$, crystallises from alcohol in slightly yellow needles, melts at 225°, and sublimes without decomposition. It is a neutral substance, and is not dissolved by acids or alkalis. It yields hemipinic acid and benzylamine when boiled for a long time with potash (sp. gr. 1·15), but when the reaction is interrupted after a short time benzylhemipinamic acid is obtained.

Benzylhemipinamic acid,

$$COOH \cdot C_6H_2(OMe)_2 \cdot CO \cdot NH \cdot C_7H_7$$ [COOH : CONHC$_7$H$_7$ = 2 : 3,

or 3 : 2 and (OMe)$_2$ = 4 : 5], crystallises from alcohol in very slender needles, and is, as a rule, more readily soluble than the imide. It loses water when heated, and is converted into the imide, so that it has the same melting point as the latter. It is readily soluble in alkalis and ammonia, but when an ammoniacal solution is evaporated the imide is obtained. The *potassium* salt crystallises in white needles, and is readily soluble in water. The *silver* salt is amorphous. The *lead* salt crystallises in microscopic, rhombic plates. The *calcium* salt, $(C_{17}H_{16}NO_5)_2Ca$, crystallises in small needles, and is rather easily soluble in water.

Veratric acid and papaveraldine, together with small quantities of acetic and oxalic acid, are obtained when papaverine ethobromide is oxidised with potassium permanganate, and the reaction products separated as described above, but the chief product is ethylhemipinisoimide. No compound analogous to benzylpapaverine-ammonium hydroxide (or oxide) is formed.

Ethylhemipinisoimide, $C_{12}H_{13}NO_4$, crystallises from boiling alcohol, in which it is only sparingly soluble, in yellowish needles, melts at 226—227°, and sublimes in colourless needles.

Ethylhemipinamic acid, $C_{12}H_{15}NO_5$, is formed when the preceding compound is heated with potash for a short time. It loses water when heated, and melts at the same temperature as the imide. The salts are similar to those of the corresponding benzyl-derivative.

Hemipinic acid and ethylamine are obtained when ethylhemipinisoimide is boiled for a long time with potash. Hempinic acid and cinchomeronic acid are formed when dimethoxyquinoline (compare Goldschmiedt, this vol., p. 302) is oxidised with 4 per cent.

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alkaline potassium permanganate. This dimethoxy-compound is therefore a derivative of isoquinoline.

The acid described as dihydroxycinchonic acid (Abstr., 1886, 478) yields a yellow, crystalline substance, when heated above its melting point until gas is no longer evolved; the product is sparingly soluble in water, melts at 230°, and gives a reddish-violet iron reaction. It yields isoquinoline when distilled with zinc-dust.

F. S. K.

Constitution of Papaverine. By G. Goldschmiedt (*Monatsh.*, **9**, 349—360).—Papaverine is a derivative of isoquinoline; it contains the two groups, dimethoxyisoquinoline, $<^{N:CH-}_{CH:CH}>C_6H_2(OMe)_2$ $[(OMe)_2 = 1:2 \text{ or } 3:4]$, and dimethylhomocatechol, which are most probably united by the CH_2-group of the latter. The constitution of papaverine is therefore probably $C_6H_3(OMe)_2\cdot CH_2\cdot C<^{C_6H_2(OMe)_2}_{N\cdot CH:CH-}>$, $[(OMe)_2 = 1:2 \text{ or } 3:4]$, and in accordance with this view papaveraldine has the constitution $C_6H_3(OMe)_2\cdot CO\cdot C<^{C_6H_2(OMe)_2}_{N\cdot CH:CH-}>$, and papaverinic acid the constitution

$$C_6H_3(OMe)_2\cdot CO\cdot C<^{C(COOH)}_{N\cdot CH:CH}>C\cdot COOH.$$

F. S. K.

Cocaïne and its Salts. By B. H. Paul (*Pharm. J. Trans.* [3], **18**, 781—785).—Ready crystallisability was pointed out by Lossen and Wöhler as a characteristic property of pure salts of cocaïne, and this is confirmed by the author, who also rejects Stockman's hypothesis that the uncrystallisable portion of cocaïne hydrochloride is a solution of cocaïne in hygrine. Hygrine he regards as a mythical substance, contending that the true explanation is, that an amorphous alkaloïd is associated with cocaïne in the coca leaves. He has obtained this alkaloïd in quantity sufficient for a detailed examination, and in the meantime states that it has a yellow colour and a bitter taste, and that it produces only a very slight anæsthetic effect on the tongue, but not until after the lapse of several minutes. Its hydrochloric acid solution on evaporation leaves a gummy residue without any sign of crystallisation, and ammonia, whilst failing to produce in it the precipitate of needle-shaped crystals characteristic of pure cocaïne, causes the liquid to become milky, and soft clots to separate. The amorphous alkaloïd has a peculiar smell like benzoïn and butyric acid, and when it is heated with hydrochloric acid in a sealed tube benzoic acid is formed.

R. R.

Ptomaïnes. By O. de Coninck (*Compt. rend.*, **106**, 1604—1605).—The ptomaïne $C_5H_{11}N$ (this vol., p. 730) forms two mercurochlorides, one of which, $(C_5H_{11}N,HCl)_2,HgCl_2$, crystallises in small, white needles slightly soluble in water and in dilute alcohol, but insoluble in absolute alcohol. It decomposes when exposed to moist air. The other is a sesqui-salt, $(C_5H_{11}N,HCl)_2,3HgCl_2$, and forms long, yellowish

needles, which are insoluble in the ordinary solvents, and decompose when exposed to moist air.

The methiodide crystallises in slender, white needles, which alter very slowly when exposed to the air, and dissolve in all proportions in absolute alcohol. With alcoholic potash, it gives a deep red coloration, which is intensified by hydrochloric or acetic acid, but is destroyed by a slight excess of ammonia. The liquid acquires no fluorescence. If the methiodide is heated with solid potash and a small quantity of water, a garnet-red coloration is produced, and on more strongly heating, an odour of pyridine dihydride is evolved. If the methiodide is distilled with aqueous potash, the distillate has a red colour, and when exposed to the air becomes highly fluorescent. In some cases, the red coloration rapidly changes to dull green, owing to the reducing action of the ptomaïne which is liberated towards the end of the process. Only a very slight excess of ammonia is required to destroy the red coloration, and in this respect the ptomaïne differs from the methiodides of the coal-tar pyridines and of the bases obtained from cinchonine and brucine. It is evident, however, that in its general behaviour this ptomaïne resembles the pyridine bases. C. H. B.

Alkaloïds in Human Urine. By J. L. W. Thudichum (*Compt. rend.*, **106**, 1803—1806).—The urine was mixed with 5 per cent. of sulphuric acid previously diluted with twice its own volume of water, and the alkaloïds were precipitated by phosphomolybdic or phosphotungstic acid. The precipitate was washed, treated with barium hydroxide and barium carbonate, care being taken to avoid an excess of hydroxide, and the deep red solution thus obtained was filtered. If ferric chloride is added to the red liquid, it produces a bulky precipitate, which contains *urochrome*, the colouring matter of the urine in combination with iron. This urochrome may be isolated in several different ways, and then treated with sulphuric acid, or the precipitate may be treated directly with the acid. In either case, the product answers to the description given by Proust in 1881. It is a deep, violet-red, bulky precipitate, which when treated with ether yields a resin and a mixture of omicholin and omicholic acid. The portion insoluble in ether consists of a red compound, *uropittin*, soluble in alcohol, and a black resin, *uromelanin*.

Omicholin has approximately the composition $C_{24}H_{38}NO_5$, and is a red, resinous substance, insoluble in ammonia, but soluble in ether and alcohol. Its solution shows a bright green fluorescence, and gives an absorption-spectrum consisting of a band between D and E.

Omicholic acid has the composition $C_5H_{22}NO_4$, and is also a resinous, red substance soluble in ether or alcohol, forming a solution which shows a green fluorescence and gives an absorption-band between D and E. This band is, however, narrower than the band given by omicholin. Omicholic acid is soluble in ammonia, and is reprecipitated by acids.

Uropittin was not obtained pure. It is always mixed with one or other of its modifications, *meta-uropittin* and *uro-rubin*, and is partially altered by contact with the oxygen of the air. It contains

11 per cent. of nitrogen. Its alcoholic solution is red, and gives an absorption-band at F.

Uromelanin has the composition $C_{36}H_{43}N_7O_{10}$, and is insoluble in alcohol or ether, but dissolves in dilute solutions of the alkalis, from which it is precipitated by acids. With silver, barium, calcium, lead, and zinc it forms basic and acid salts. The silver salt has the composition $C_{36}H_{40}AgN_7O_9$. Uromelanin is a very stable substance; the quantity excreted by an adult is 0·3 to 0·5 gram per day.

Neither urochrome nor any of the other products can be obtained crystallised. Urochrome is an alkaloïd, the function of which is as yet unknown. The products of its decomposition are not related to the colouring matters of the blood or of the bile.

If the filtrate from the urochrome iron precipitate is concentrated, it yields bulky crystals which may be purified by recrystallisation from alcohol. These consist of an alkaloïd, *uro-theobromine*, isomeric with ordinary theobromine. It sublimes without change, forms no crystalline precipitate with silver nitrate, and displaces acetic acid from cupric acetate, forming an insoluble compound.

Creatinine is also present, and the mother-liquor from the creatinine contains three alkaloïds. *Reducine*, $C_{12}H_{26}N_6O_9$ or $C_6H_{11}N_3O_4$, forms a barium compound which is almost insoluble in alcohol. Neutral or acid solutions of reducine reduce ferric, cupric, or mercuric salts to ferrous, cuprous, or mercurous salts respectively, and silver salts to metallic silver. *Para-reducine* unites with zinc oxide to form a compound, $C_6H_9N_3O{\cdot}ZnO$ or $C_6H_9ZnN_3O_2$. *Aromine* could not be isolated in a pure condition. When heated, it gives off an aromatic odour resembling that obtained from tyrosine under similar conditions.

C. H. B.

Oxidation of Albumin with Permanganate. By R. Maly (*Monatsh.*, **9**, 255—283; compare Abstr., 1885,824).—The author has previously described an oxyprotosulphonic acid obtained by oxidising albumin with permanganate. On further treatment with permanganate, this acid is converted into *peroxyprotoïc acid*. Like all albuminous bodies it gives the biuret reaction, contains sulphur, and its carbon and nitrogen are present in the same ratio as in albumin, from which it differs in not being precipitated by a solution of potassium ferrocyanide containing a little acetic acid, or by tannic acid. Baryta readily decomposes the acid with formation of the following substances: — Oxalic, sulphurous, isoglyceric, amidovaleric, benzoic, formic, and glutamic acids, leucine, ammonia, and traces of pyrroline.

G. T. M.

Physiological Chemistry.

Coagulation of the Blood. By J. B. Haycraft and E. W. Carlier (*Brit. Med. Jour.*, **2**, 1888, 229—232).—The blood was received directly from the finger-tip into a tall, cylindrical vessel filled with viscid castor-oil. By occasionally inverting the vessel the blood may be kept from coming in contact with the sides or ends of the vessel for a considerable time. Blood removed in this way from the body remains uncoagulated for an hour or more. Not only is its fluidity preserved, but the white corpuscles remain rounded in shape, and the blood plates do not tend to run together or change in any way. As soon, however, as the blood comes in contact with solid matter, other conditions remaining the same, the white corpuscles exhibit amœboid movement (if the temperature is high enough), fibrin is formed, and the blood plates become adhesive. The conclusion is drawn that all these changes result from the mechanical stimulation of living and naked protoplasm by solid matter. Actual disintegration leading to the disappearance of the white corpuscles was not observed, but the following were the appearances noted:—The coarsely granular corpuscles became flattened and irregular in outline, due to amœboid movement. They then lost their granules, or these retired to one part of the cell, the rest of the cell becoming clear. The strings of fibrin would then be seen gradually forming. The finely granular corpuscles were observed to present the same appearance, with the exception that their fine granules did not congregate to such an extent. The production of fibrin ferment from the white corpuscles, and probably also from the blood plates, which are regarded as pieces of undifferentiated protoplasm, is regarded not so much as a result of death and disintegration, as of living metabolism in those structures. W. D. H.

Alcaptonuria. By R. Kirk (*Brit. Med. Jour.*, **2**, 1888, 232—233).—A certain kind of human urine darkens on the addition of alkalis. Bödeker (*Annalen*, **117**, 98) isolated from such urine a substance to which he gave the name "alcapton." Ebstein and Müller (*Virchow's Archiv*, **62**, 554) found, as they supposed, catechol, and Smith (*Dublin Med. Jour.*, **73**, 465) protocatechuic acid. In a former paper (*Brit. Med. Jour.*, **2**, 1886, 1017), the author showed that the substance differs somewhat in its reactions from both those compounds, and gave it the name urrhodinic acid. Further investigation has shown that a mixture of two substances is in reality present, one which is precipitable by neutral lead acetate, and one which is not. The former is called *uroleucic acid;* this is soluble in water, alcohol, and ether; it crystallises from ether in stellate groups; it has a strongly acid reaction, reduce Fehling's solution and Löwe's bismuth solution. The crystals melt at 133·3°, and have the formula $C_9H_{10}O_5$. A solution gives with ferric chloride (1 in 40) a transient, green colour; a drop of the ferric solution added to the crystals produces a red colour.

The substance which is not precipitated by lead acetate is amorphous and has an acid reaction. It exercises the same general reducing reactions, but these are not so keen, and in fact it corresponds in all its properties with Bödeker's alcapton. It is provisionally termed uroxanthic acid. W. D. H.

Action of Artificial Dyes on the Animal Organism. By T. Weyl (*Ber.*, **21**, 2191—2192).—Naphthol-green B has no injurious effects on dogs when administered directly into the stomach in daily doses of 1 to 2 grams for 14 days. Dinitrocresol (compare this vol., p. 520) is poisonous. Martius'-yellow (dinitro-α-naphthol), which is not fatal to rabbits (compare this vol., p. 184), is poisonous to dogs of medium size, even in small doses. 0·5 gram was injected into the stomach of a dog weighing 6850 grams, on two consecutive days, and on the third day 1 gram of the sodium salt; the animal died after the last injection.

Subcutaneous doses of 0·1 gram daily for four days, or two doses of 0·2 gram, killed a dog weighing 8800 grams. Naphthol-yellow S (sulphonic acid of Martius'-yellow) is quite uninjurious even in doses four times as large as fatal doses of Martius'-yellow. Aurantia is probably poisonous, but up to the present time no azo-dye is known to be poisonous. Three commercial samples of safranine proved fatal when administered in three subcutaneous doses of 0·5 gram of the hydrochloride to dogs weighing 5 to 6 kilos. F. S. K.

Chemistry of Vegetable Physiology and Agriculture.

Bacteriological and Chemical Investigation of some Bacilli. By J. Kunz (*Monatsh.*, **9**, 361—377).—*Bacillus strumitis* was found by Tavel in two cases of *struma cystica*, both patients having suffered intense intestinal catarrh; it also occurs in animals. Its length varies from 1 to 1·5 μ, and its breadth from 0·5 to 0·6 μ. It thrives well in Koch's peptone gelatin; in agar-agar jelly—especially in presence of sugar, growth is very rapid, and a large quantity of gas is evolved, but in no case is any particular odour perceptible. It is extremely mobile, and is best stained with alkaline methylene-blue by Löffler's method. When 140 grams of inoculated gelatin, containing 2 per cent. of grape-sugar, are kept for three days at the ordinary temperature, and finally for 12 hours at 32°, 0·0149 gram of pure carbonic anhydride is evolved. Lactic acid and succinic acid are produced when it is grown in nutrient gelatin containing 5 per cent. of grape-sugar. 1500 grams of the solution gave 1·57 gram of succinic acid and 1·77 gram of zinc lactate. It grows in milk without causing coagulation or change of reaction.

Bacillus pyocyaneus, the cause of the colour in blue and green pus, is 3·2 μ long and 0·8 μ broad on the average. Inoculated gelatin kept for three to four days at the ordinary temperature, and then for seven

days at 35°, liquefies in a few days, shows a green fluorescence, and has the specific smell of blue pus. Pyocyanin and pyoxanthose were both isolated from the solution, but the liquid still showed a green fluorescence due to a distinct colouring matter which is only soluble in water and alcohol, and is not destroyed by boiling. Concentrated solutions of this colouring matter transmit red and green light only, but dilute solutions have no absorptive power. Only a few centigrams of pure pyocyanin were obtained from 6 kilos. of the solution; it contains nitrogen and sulphur, and is not hygroscopic. The green pigment which is formed when this bacillus is grown in nutritious gelatin is very probably produced by the oxidising action of the air on a chromogen which is formed by the bacillus, as the pigment is not contained in the bacterial cells. In gelatin solutions, the green colour disappears gradually at the ordinary temperature in 10 to 15 weeks, giving place to a dark, reddish-brown shade, and the reaction becomes strongly alkaline. The bacillus grows in milk, and produces a yellowish-green solution which becomes intensely green when ammonia is added.

The cells of *Bacterium phosphorescens* are almost circular, being 1·3 to 1·9 μ long and 1·1 to 1·7 μ broad; each cell is mobile and surrounded by a clearly perceptible zoogloea membrane; sometimes two are united. It grows slowly at the ordinary temperature in peptonised gelatin or in peptonised gelatin containing 2 per cent. of grape-sugar, but only at the surface, and the property of emitting light seems to be dependent on the presence of oxygen. It grows well in 2, 3, and 4 per cent. solutions of sea-salt, containing 0·25 per cent. of peptones, forming thick rods 2 to 2·9 μ long and 0·9 to 1·2 μ thick on the average. These solutions are very phosphorescent, far more so than any inorganic substances or an alkaline amyl alcohol solution of lophin; on shaking, the phosphorescence becomes more clearly apparent, but on cooling to 0° its intensity is slightly diminished. The phosphorescence disappears when the solution is heated at 35° for a few minutes, but reappears on cooling; it is, however, completely destroyed by heating at 35° for 15 minutes. After two to three weeks, the solutions become yellowish and gradually lose their phosphorescence; after several weeks, phosphorescence ceases entirely, but the bacteria do not die. The solutions phosphoresce equally well if placed in the dark immediately after inoculation. The phosphorescence is most probably caused by some vital process, as it is destroyed by all reagents which kill the protoplasm of the cells.

Bacterium phosphorescens grows in 3 per cent. solutions of sodium chloride, magnesium sulphate, or sodium sulphate containing 0·25 per cent. of peptones, and the solutions show intense phosphorescence. It also grows in urine and in milk.

Koch's comma bacillus grows very quickly in a solution prepared from serum albumin and fresh pancreatic glands, and the solution soon becomes turbid. 3 litres of such a solution were kept for three days at 35°, and then treated by Brieger's method. 0·01 gram of the ptomaïne obtained was injected subcutaneously into a frog, but without any apparent result; the same quantity injected into a mouse proved fatal in two hours. A subcutaneous dose of 0·02 gram was

given to a rabbit weighing 1100 grams; after two hours, the action of the salivary glands was greatly stimulated, and diarrhœa set in, but the animal ultimately recovered. A non-poisonous base was separated from the ptomaïne; its platinochloride, $(C_2H_5N)_2,H_2PtCl_6$, is sparingly soluble in water, and when treated with soda gives off a smell very similar to that of human semen. (Compare Schreiner, Abstr., 1879, 72, and Ladenburg and Abel, this vol., p. 441.)

Small quantities of succinic acid are formed by the growth of the comma bacillus, but lactic acid is not produced. F. S. K.

Cultivation of Bacillus Lepræ. By B. Rake (*Brit. Med. Journ.*, **2**, 1888, 215—221).—As a result of numerous experiments carried out at the Trinidad Leper Asylum, the following conclusions are drawn:—

1. At a tropical temperature, *Bacillus lepræ* does not grow on the ordinary nutrient media.

2. In all animals yet examined, inoculation or feeding with leprous tissues is not followed by local growth or general dissemination of the bacillus.

3. When the bacillus is placed in putrid fluids or buried in the earth, it also does not multiply. W. D. H.

Unicellular Algæ. By V. Jodin (*Ann. Agronom.*, **14**, 241—255).—The author has made cultivations of various species of *Protococcus*, *Zygnema*, &c., in artificial media, consisting of solutions of the requisite minerals in distilled water. The most suitable solution is the same as that used by Raulin in his experiments on *Aspergillus niger*. The solution is placed in flasks which are exposed to the light; the carbonic anhydride is renewed in the air of these flasks by an automatic generator. This simply consists of a flask filled with a solution of ferric oxalate, connected with the culture flask by a bent glass tube passing through the caoutchouc stopper of the latter. The ferric oxalate evolves carbonic anhydride on exposure to light. Under favourable circumstances the crop obtained in several weeks' exposure amounts to 10 grams of fresh algæ or 1 to 2 grams of dried product per litre. These cultivations are well adapted to throw light on the chemical processes taking place in the green cell, since the crops obtained are uniform and homogeneous and free from the disturbing influences arising from the differentiation of organs and the migration of proximate principles in the higher plants. Moreover these algæ live completely immersed in water. As regards phosphoric acid, the author finds that its percentage varies from 0·5 to 3·0 of the dry matter, being greatest when the nutritive medium is rich in phosphoric acid. Although the living cell has the power to exhaust the solution of phosphoric acid, yet the same cell dried over sulphuric acid, and consequently with its physiological activity impaired or destroyed, yields up to water in which it is macerated nearly all the phosphoric acid previously assimilated. The proportion of nitrogen in *Protococcus* varies from 1·43 to 6·67 per cent. of the crop. The conditions of assimilation of this element are still under experiment.

J. M. H. M.

Influence of Compressed Oxygen on the Growth of Plants. By S. JENTYS (*Ann. Agronom.*, **14**, 275—278).—The experiments were made in an apparatus belonging to the University of Tubingen, and the gases, after being carefully purified, were compressed by the aid of the town water supply which is delivered at 7·5 atmospheres. Young plants of beans, sunflower, peas, buckwheat, radish, &c., were chosen, the length of the shoot above a fixed mark carefully noted, and the subsequent growth in the gases employed measured or observed through a horizontal microscope. Errors due to heliotropic curvature were avoided by surrounding the apparatus with a black curtain. The general results are that growth is in no case completely arrested by increase in the pressure of oxygen above the atmospheric pressure, but it is always retarded, and the more so the greater the pressure, provided the duration of the experiments is the same. Peas are more sensitive than beans, and beans than sunflowers. As regards pressures equal to or less than the atmosphere, the augmentation of the partial pressure of oxygen up to one atmosphere is decidedly favourable to the growth of the radish, white mustard, and turnip; it is almost without influence on that of the bean, sunflower, and pea. Carefully purified oxygen at atmospheric pressure shows no injurious influence at the end of three days' exposure. Compressed air retards growth, but the retardation is due to increased pressure of the nitrogen, since plants grow more slowly in air compressed to 5 atmospheres than in pure oxygen at the atmospheric pressure. The influence of mixtures of compressed hydrogen and nitrogen with oxygen at its normal pressure in the air is always unfavourable. Respiration and growth are in general affected both in the same direction by alteration of pressure. The injurious influence of oxygen above atmospheric pressure is not due to any inability to support respiration, but to some actively poisonous influence exerted after a time by the gas at high pressures. J. M. H. M.

The Rôle of Oxygen in Plant Life. By W. PALLADIN (*Ann. Agronom.*, **14**, 278—279, from *Bull. Soc. Imp. Nat. Moscow*, 1886, 44—133).—The principal sign of fermentation, or life without oxygen, is the greater expenditure of material than takes place under normal circumstances in the air. Experiments made by the author on the young roots of germinating beans bring the intramolecular respiration of the higher plants under this formula. The cuttings of roots 2 centimetres in length lost 4·6 per cent. of their dry weight during normal respiration in the air for 20 hours, and more oxygen was absorbed than was given out again as carbonic anhydride. Placed for 20 hours in a gas deprived of oxygen, the same roots lost 11 per cent. of their weight, in spite of the gradual slackening of the vital phenomena. The carbonic anhydride evolved was 0·55 of the loss of weight, proving that this gas is not the sole volatile product of intramolecular respiration. J. M. H. M.

Decomposition of Carbonic Anhydride by Plants deprived of Chlorophyll. By HUEPPE (*Ann. Agronom.*, **14**, 274).—In this note, the idea is advanced that certain plants deprived of chlorophyll,

such, for example, as one of the nitrifying (?) bacteria mentioned by Heraeus, are capable of decomposing carbonic anhydride, and employing the carbon for the synthesis of carbohydrates. For example, carbonate of ammonia is stated to be decomposed by the bacterium in question with formation of ammonia, aldehyde, and nascent oxygen. The aldehyde is built up into carbohydrates, and the nascent oxygen nitrifies the ammonia to nitric acid. J. M. H. M.

Formation of Starch by Plants from Organic Solutions. By E. Laurent (*Ann. Agronom.*, **14**, 273—274).—The author's results on this subject agree with those of A. Meyer, and were obtained during the same year. He employed etiolated shoots of potato plants in which the reserve material was exhausted, and not a trace of starch could be found in sections taken at different heights. They were plunged in solutions of a great number of different organic compounds, of degrees of concentration varying from 0·2 to 20 per cent., and placed in a dark place. Only seven compounds were transformed into starch, namely, glycerol, dextrose, lævulose, galactose, saccharose, lactose, and maltose. Only in very rare cases was there any growth without formation of starch. Whether any of the substances were utilised for respiratory purposes without either formation of starch or growth of the stem, is an undetermined point. J. M. H. M.

Formation of Organic Acids in Growing Plants. By W. Palladin (*Bied. Centr.*, 1888, 499).—The author shows that during the exhalation of plants, the ratio $CO_2 : O_2$ is less than unity; and he calculates, using Lieberkühn's formula, that when all the nitrogen of the albumin has been converted into asparagine that there remains a residue containing no oxygen; this residue must either be oxidised, or else must be absorbed in the formation of cellular tissue, but in either case this change must be accompanied by a strong assimilation of oxygen whereby $CO_2 : O_2$ becomes less than 1. By the transformation of asparagine and carbohydrates into albumin, a strongly oxidised residue must remain, and acids are a bye-product from such regeneration; it is also considered that the water produced by exhalation is a product of regeneration. E. W. P.

Formation of Crystals of Calcium Oxalate in Plant Cells. By J. H. Wakker (*Ann. Agronom.*, **14**, 274—275, from *Bot. Centr.*, **33**, 360).—According to the author, the crystals are not formed in the mass of protoplasm, but in the cell sap, and are always found in the vacuoles. J. M. H. M.

Influence of Magnesium and Calcium Chlorides on Germination. By R. Hindorf (*Bied. Centr.*, 1888, 461—462).—The injury which follows the application of Stassfurth waste salts to plants arises from the chlorine present, but then only when the salts are in too great quantity. Germination of wheat, rye, barley, oats, peas, &c., is assisted by these salts. The sulphates also are injurious, but not to such an extent as the chlorides. E. W. P.

Glutinous Rice of Japan. By SHIMOYAMA-YUNICHIRO (*Ann. Agronom.*, **14**, 287—288, from *Bot. Centr.*, **32**, 6).—There are many varieties of glutinous rice in Japan. Besides ordinary starch, the starch of the Mozi rice consists of soluble starch, dextrin, and perhaps maltose,—that is, products which are generally obtained from starch by artificial processes. The ordinary starch can only be recognised by the iodine reaction after the extraction of the greater part of the dextrins. The other cereals cultivated in Japan also contain starch similar to that of the Mozi rice. The starch of the potato, and ordinary starch yield soluble starch, or at least a body coloured red by iodine, and also dextrin. The difference between all these varieties of starch is therefore quantitative not qualitative. The Mozi rice contains too little ordinary starch to form a paste, or to give a yellow colour with bromine. It contains no substance giving a violet colour with iodine as Nägeli supposes. J. M. H. M.

Absorption of Nitrogen by Soils and Plants. By A. GAUTIER and R. DROUIN (*Compt. rend.*, **106**, 1605—1607).—A reply to Chevreul (this vol., p. 979).

Experimental Plots at the Agricultural Station of La Somme. By A. NANTIER (*Ann. Agronom.*, **14**, 255—261).—*Wheat.*—Experiments were made with six well-known varieties of wheat at Senarpont on two very different soils, the one clayey and the other calcareous. In the rich clay, all gave good crops, Dattel and Bordeaux the best; in the light soil, Bordeaux was decidedly the best; in both soils, the Browick variety answered poorly. One result of these experiments is that the highly cultivated varieties need good, deep, rich, well-prepared soil. Various artificial manures were also tried with the Bordeaux wheat on the two soils already mentioned. The phosphatic manures were applied in the autumn, the soluble manures in the spring. The clay soil contained 0·207 per cent. N, 0·134 per cent. P_2O_5, and 0·237 per cent. K_2O; the calcareous soil, 0·202 per cent. N, 0·148 per cent. P_2O_5, and 0·149 per cent. K_2O. Although these chemical features of the soils were so similar, the action of manures was very different in the two cases. None of the manures employed gave any increase in the calcareous soil, whilst on the clay soil sodium nitrate, ammonium sulphate, soluble and insoluble phosphate each gave an increase of about 10 quintals per hectare, the unmanured plot yielding 30 quintals. Potassium salts alone gave no appreciable increase, as might have been predicted. The soluble and insoluble phosphates were equally efficacious.

Irrigated Pastures.—Figures cited by the author show that a pasture which without irrigation gave 1500 kilos. hay per acre as first cut, and no aftermath, when irrigated gave 5000 and 6000 kilos. hay first cut, 2700 and 3500 kilos. second cut, total 7700 and 10,100 kilos.

Insoluble Phosphates.—These must be well worked into the soil in order to produce good results. Applied only on the surface of a pasture poor in phosphoric acid (0·149 per cent. P_2O_5) but rich in nitrogen (0·481 per cent.) and potash (0·138 per cent. K_2O), little or no effect was produced. J. M. H. M.

Richness and Density of Wheat. By A. PAGNOUL (*Ann. Agronom.*, **14**, 262—272).—The average composition is as follows:—Water, 14·3; albuminoïds, 11·0; starch and dextrin, 70·5; fatty matters, 1·2; cellulose, 1·7; ash, 1·6. The water being always near 14·0, and the sum of the ash and cellulose 3 to 4, in order to arrive at the starch, dextrin, and fat, it suffices to add 17 to the albuminoïds, and subtract the sum from 100. The albuminoïds are very variable, and in order to correlate the variation in this constituent and in the phosphoric acid with the physical characters of the grain, the author has examined 70 samples of different countries, varieties, and methods of growth, and has embodied the results in an elaborate table, under the following heads, arranging the different samples in order of their richness in nitrogen:—

Water.—This varies from 10·00 to 16·88 per cent.; mean 14·1.
Albuminoïds, in undried sample, 7·87 to 16·06 per cent.; mean 10·43.
Albuminoïds, in dried sample, 9·13 to 17·87 per cent.; mean 12·14.
P_2O_5, in undried sample, 0·448 to 1·016 per cent.; mean 0·661.
P_2O_5, in dried sample, 0·533 to 1·154 per cent.; mean 0·769.
Number of grains in 10 grams, 162 to 404; mean 240.
Weight of 1 grain in milligrams, 24 to 61; mean 41.
Volume of 1 grain cubic millimetres, 18 to 46; mean 32.

He also gives the apparent and real densities of the undried grain and the real density of the dried grain (calculated).

The richness in nitrogen and the density generally increase together, but not in any constant ratio. The samples rich in nitrogen are usually rich in phosphoric acid also, but here again the relation is an irregular one. There is no constant relation between the size of the grains and their richness in nitrogen, but usually the small grained varieties are richer than the large ones. The richness in nitrogen appears to depend more on the mode of cultivation and meteorological conditions than on the variety; different samples of the same variety are found in all parts of the tables. The different coloured grains in the same sample differ much in composition; thus in a sample of Berques wheat, the white grains gave 10·22 per cent. of albuminoïds (dry), and the red grains 13·87 per cent. Tropical wheats are not always the richest in albuminoïds; two samples from Egypt were amongst the poorest, whilst the red wheat of Saint Pol (France) was one of the richest. The square-headed Porion wheat, from which large yields have been obtained, gave 11·87 per cent. of albuminoïds in the dried sample, close on the average of the whole series. J. M. H. M.

Application of Potassium Chloride to Sugar Beet on Heavy Soil. By A. PETERMANN (*Bied. Centr.*, 1888, 443—450).—The results of three years' experiments show that when potassium chloride is applied as a manure to sugar beet, in conjunction with nitre and superphosphate, an increase in total yield is produced in some cases, yet the absolute yield of sugar per hectare is reduced.

E. W. P.

Analytical Chemistry.

Spectroscopic Notices. By H. W. Vogel (*Ber.*, **21**, 2029—2032). —*Coal-gas Volatiliser.*—A test-tube containing the substance under examination is fitted with a double-bored cork. Into the one cork a tube is fitted which passes nearly to the bottom, whilst into the second hole a tube is fitted bent twice at right angles, and drawn out at the upper end to a jet. Above the jet a tube 10 cm. long and 57 mm. wide is held by means of a wire in such a way that it can be pushed down over the jet or drawn up as required. Coal-gas is passed through the apparatus over the heated solution in the tube, and burns at the jet. The wide tube is so arranged that the flame is non-luminous. The apparatus can be used in most cases where the "hydrogen volatiliser" has hitherto been used, and is more convenient.

2. *Detection of Chromates.*—This is most readily effected by heating the substance in the apparatus just described with sodium chloride and sulphuric acid, when chromium oxychloride is formed if a chromate is present. A very characteristic spectrum is obtained, and 1 mgram. of a chromate can be detected.

3. *Observations of Deep Layers of Liquids in Test-tubes.*—The apparatus employed consists of a vertical spectroscope, a test-tube containing the liquid to be examined, and below this a mirror to reflect light through the liquid in the test-tube. In order to get over the inconvenience caused by the round bottom of the test-tube containing the liquid acting as a lens which focuses the light reflected into the tube to a point within the liquid, the test-tube is fitted into a larger test-tube which contains a little water, and thus forms a plano-convex lens. The outer tube being a fixture, the inner tube is pushed up or down until the greatest amount of light is obtained. With a depth of 16 cm., the chromous reaction can be observed when only 1·5 per thousand is present. Sketches of the apparatus are given.

N. H. M.

Funnel for Filtering Carbon. By T. M. Drown (*Chem. News*, **57**, 223).—This funnel has a cylindrical stem, of which the diameter is slightly less than the internal diameter of the porcelain combustion tube used. In use, the funnel is about half filled with asbestos, which is supported by a flat coil of platinum or copper wire, the end of which extends from the centre to below the end of the stem. When all the carbon is on the filter, the coil is withdrawn through the stem, and the funnel and contents dried. Now by inserting the stem in the combustion tube, the contents are easily and safely transferred. Successful determinations have been made by treating iron borings directly on the filter with cupric ammonium chloride solution, washing with dilute hydrochloric acid, &c., and burning without removal of the small amount of copper which may be present. Direct treatment in a glass combustion tube is not as satisfactory as using the funnel and transferring to a porcelain tube.

D. A. L.

Estimation of Bromine. By J. T. White (*Chem. News*, **57**, 233—245).—Bromine is evolved when a bromide is heated with potassium permanganate and aluminium sulphate, whilst chlorides and iodides do not yield chlorine or iodine under these conditions. The author proposes a method of estimating bromine. The solution of a bromide is mixed with 10 c.c. of a permanganate solution (1 in 25) for each 0·1 gram bromine, and then with some aluminium sulphate solution saturated at ordinary temperatures; the bromine is distilled off in a current of carbonic anhydride and titrated.

D. A. L.

Determination of Phosphorus in Iron by Molybdate. By C. Meineke (*Zeit. angewand. Chem.*, **1**, 68—72).—The author cannot confirm Tamm's statement (this vol., p. 529) that the presence of manganese is prejudicial to the determination of phosphoric acid. He obtains identical results whether the iron is separated from the manganese as acetate, or whether the treatment is omitted. The amount of phosphoric acid precipitable after simple dissolution of the iron in nitric acid does not bear any constant proportion to that found after evaporation and ignition. Instead of the evaporation and ignition, the complete oxidation of the phosphorus can be effected by adding chromic acid to the nitric acid solution. With irons containing more than 3 per cent. of carbon, the addition of some sulphuric acid is necessary. The process is as follows:—4·375 grams of the iron are dissolved in 50 c.c. of nitric acid; 30 c.c. of diluted sulphuric acid (equal volumes of strong acid and water) is added, and the whole is evaporated to 15 or 20 c.c.; 2½ or 3 grams of chromic acid is now added, the liquid boiled for 10 minutes and then diluted. If manganese peroxide separates, it must be reduced by hydrogen dioxide, but this generally introduces traces of phosphoric acid. The solution is now made up to 250 c.c., filtered, and 100 c.c., after partial neutralisation with ammonia and heating to 90°, is precipitated with molybdate. The precipitate after being washed, first with acidified ammonium nitrate, and then with cold water (see this vol., p. 194), is gently ignited. A gram corresponds to 1 per cent. of phosphorus in the iron.

The organic matters in bog-iron ore are without influence on the determination of the phosphorus it contains. M. J. S.

Determination of Alkaline Hydroxides in Presence of Carbonates. By A. Isbert and Venator (*Zeit. angewand. Chem.*, **1**, 109—110).—The perfectly cold solution of the mixture is titrated with standard acid, using as indicator the special rosolic acid prepared as described in *Zeit. anal. Chem.*, 1887, 583. The change to yellow is perfectly distinct and sharp as soon as the hydroxide is neutralised. On now boiling the liquid, the red colour is restored, and a further quantity of acid, corresponding with the carbonate present, is required to produce the change to yellow. M. J. S.

Volumetric Estimation of Potassium and Sodium. By J. T. White (*Chem. News*, **57**, 214—215).—21 c.c. of the solution containing about 0·2 gram of the mixed chlorides is placed in a 100 c.c.

flask, and for each 0·1 gram of the mixed salts 5 c.c. of a solution of ammonium hydrogen tartrate is added. This solution is prepared by diluting a solution of ammonium hydrogen tartrate saturated at 90° with an equal volume of water, and warming before use to dissolve any crystals. When the flask and contents are cool, methylated spirit is added gradually, shaking after each addition until the flask is filled to the mark; after three hours or longer, the potassium hydrogen tartrate is filtered off, and 10 c.c. of the solution evaporated to dryness, ignited, then again evaporated with ammonium chloride solution, and re-ignited. The residual sodium chloride is titrated with silver nitrate. D. A. L.

Solubility of Magnesium Ammonium Phosphate in Alcohol. By A. J. Wakemann (*Technology Quarterly*, Boston, **1**, 173—177).—The author mentions that Rose, in the sixth edition of his *Handbuch der Analytischen Chemie*, points out that the precipitate of magnesium ammonium phosphate is entirely insoluble in water containing one-fourth of its bulk of ammonia solution of specific gravity 0·96°, and that under the determination of phosphoric acid by magnesium salts, Rose states that water containing 3 per cent. of ammonia gas dissolves only traces of the precipitate, and that this solubility is reduced to less than half if to the dilute solution of ammonia one-fourth of its volume of alcohol is added, and, further, that the addition of alcohol favours the separation of the precipitate.

The author, in order to test the method, which he says appears to have been neglected by analysts, has made a series of comparative analyses under identical conditions with the exception that some of the precipitates were washed with ammonia solution, according to the ordinary method, and the remainder with ammonia solution containing alcohol.

The results of his experiments point to a slight advantage in the use of alcohol in diminishing the solubility of magnesium ammonium phosphate when the precipitate is somewhat bulky. The use of alcohol, moreover, appears to make the precipitate more compact, so that it is more easily washed, and is less liable to creep up the sides of the funnel. The addition of alcohol to the solution in which the precipitation takes place is not advantageous, as it causes the precipitate to attach itself more closely to the beaker, so as to be difficult of removal, and it also retards the filtration. G. W. T.

Solvent Action of Rochelle Salt on Metallic Hydroxides. By H. N. Warren (*Chem. News*, **57**, 223—224).—The author includes the moist precipitates of the following metals in the list of hydroxides and carbonates soluble in solutions of Rochelle salt:—Copper, zinc, manganese, nickel, cobalt, iron (ferrous and ferric), chromium, aluminium, tin (stannic), antimony, bismuth, and even barium, strontium, calcium, and magnesium to a considerable extent in concentrated solutions of Rochelle salt. Cadmium carbonate is, however, insoluble. Treatment with Rochelle salt solution cannot, therefore, be safely employed for distinguishing or separating bismuth or tin from antimony, although it is frequently recommended for that purpose.

Cadmium carbonate may, however, be separated from copper carbonate by such treatment. D. A. L.

Determination of Manganese as Sulphide. By C. MEINEKE (*Zeit. angewand. Chem.*, **1**, 3—5).—Manganese sulphide when thrown down in the green form can be easily washed by decantation, and shows no tendency to pass through the filter. The green modification is readily obtained by precipitating from a boiling solution containing a large excess of ammonia. After boiling for a few minutes and allowing to subside, filtration may take place immediately. A trace of manganese remains in solution, but not more than after precipitation in the cold. It may be recovered, if desirable, by evaporating to a small bulk, acidifying, filtering from sulphur, and precipitating with bromine and ammonia. Traces also adhere to the precipitation vessel, and should be dissolved with hydrochloric acid containing bromine. The results are not influenced by the presence of large quantities of ammonium chloride or acetate, or of sodium chloride, or by dilution. Satisfactory results have been obtained in the separation of manganese, calcium, and magnesium. For weighing, the precipitate is best converted into Mn_3O_4, for which purpose ignition at a bright-red heat, with exposure to air until the weight is constant, suffices. M. J. S.

Determination of Small Quantities of Manganese in Iron rich in Silicon. By C. REINHARDT (*Zeit. angewand. Chem.*, **1**, 108—109).—Hampe's chlorate process (Abstr., 1886, 101) is unsuitable for such irons owing to the large quantity of gelatinous silica which separates during the solution in nitric acid. The following modification of Belani's method can be employed:—3 or 4 grams of the iron is covered with 30 or 40 c.c. of hydrochloric acid (1·19); some potassium chlorate is added, and after standing for some time in the cold the solution is heated and evaporated to 15 or 20 c.c. It is then diluted with cold water and filtered through a Schleicher's filter into a 500 c.c. flask, washing the silica with hot water. For each gram of iron, 5 c.c. of nitric acid (1·4) is now added. The solution is boiled, cooled, neutralised with zinc oxide until the ferric hydrate is precipitated, then made up, mixed, and filtered. Half of the filtrate is boiled with sodium acetate, bromine, and zinc oxide, and the precipitated peroxide is titrated with oxalic acid and permanganate.

M. J. S.

Determination of Iron by Nitroso-β-naphthol. By C. MEINEKE (*Zeit. angewand. Chem.*, **1**, 5—7).—The method described differs little from that of Ilinski and Knorre (Abstr., 1886, 100). The solution must not contain nitrates. It is desirable, although not necessary, that the iron should be in the ferric state. The solution should be as nearly neutral as possible. About 1 gram of nitroso-β-naphthol dissolved in 2 c.c. of 50 per cent. acetic acid is required for each decigram of iron. The precipitation is best performed in the cold. It is complete when a drop of the clear upper liquor gives a brown colour with a cobalt solution. The precipitate after having been washed with cold water, is so far dried that the rim of the filter can be

wrapped round it, and it is then very gradually heated in a large porcelain crucible until completely burnt to ferric oxide.

Cobalt and copper if present are precipitated along with the iron. Phosphoric acid appears sometimes to be completely and at other times imperfectly thrown down. M. J. S.

Determination of Alcohol. By B. Röse (*Zeit. angewand. Chem.*, **1**, 31—35).—An excess of potassium permanganate oxidises alcohol completely to carbonic acid and water when to the mixture so much concentrated sulphuric acid is added that it amounts to 40 per cent. of the whole. The author's experiments were made with absolute alcohol distilled from quicklime in an apparatus from which moist air was excluded. It boiled at 78·26° (corr.). From this, a 1 per cent. solution was made with purified water. The permanganate solution contained 10 grams in a litre, and was standardised by potassium quadroxalate, a decinormal solution of which is also required. The analytical process is as follows:—About 5 grams of the dilute alcohol is weighed in a flask; 50 c.c. of the permanganate is run in, and then 20 c.c. of sulphuric acid whilst shaking. After a few minutes, 100 c.c. of water is added, then a measured excess of the oxalate, the liquid heated nearly to boiling, and the excess of the oxalate found by permanganate. In four experiments, 100·18, 100·0, 100·24, and 100·0 per cent. of the alcohol present was indicated. M. J. S.

Estimation of Glycerol. By F. Filsinger (*Zeit. angewand. Chem.*, **1**, 123—124).—The substitution, proposed by Jolles, of potassium manganate for permanganate in the Benedict-Zsigmondy process (which is based on the oxidation of the glycerol in cold alkaline solution to oxalic acid and titration of the latter after precipitation by calcium chloride) is found to yield low and irregular results even when a large excess of manganate is employed. M. J. S.

Influence of Inactive Substances on the Polaristrobometric Estimation of Grape-sugar. By R. Přibram (*Monatsh.*, **9**, 395—405).—The rotatory power of solutions of grape-sugar containing acetone increases with the percentage of acetone, so that $[\alpha]_D = 16{\cdot}587 + 0{\cdot}026x$ where $[\alpha]_D$ is the rotation at 20° ($l = 2dm$), 16·587 is the rotation of the sugar alone in solutions containing 15·68 grams in 100 c.c., and x is the percentage of acetone. The rotatory power of sugar solutions containing acetone reaches its maximum only after a certain lapse of time, and the viscosity of such solutions increases with the percentage of acetone. These facts must be borne in mind in estimating the sugar in diabetic urine when more than 5 per cent. of sugar and 1 per cent. of acetone are present; smaller quantities have quite inappreciable influence on the results.

The rotatory power of tartaric acid is considerably diminished in the presence of acetone.

Carbamide lowers the rotatory power of grape-sugar, but only slightly; ammonium salts act similarly but to a larger extent.

8 to 10 per cent. of phosphates lowers the rotatory power slightly, smaller quantities have no appreciable effect. F. S. K.

Determination of Starch in Grain. By MONHEIM (*Zeit. angewand. Chem.*, **1**, 65—68).—The direct inversion of the starch with hydrochloric acid gives results which are too high, owing to the conversion of the cellulose into sugar (Märker, *Handb. d. Spiritusfabr.*, 4th Ed., 93). Asboth's method (Abstr., 1887, 868) gives most unsatisfactory results, concordant numbers being obtained only when the same baryta solution is used, and the results being even then in many cases incredible. The author gives the preference to Lintner's method. 3 grams of the ground grain with 50 c.c. of water are heated for four hours in a pressure bottle in a paraffin-bath at 135—140°. The solution is filtered, and after dilution to 200 c.c. is inverted by adding 20 c.c. of hydrochloric acid and heating for three hours on the water-bath in a flask with inverted condenser. The solution is netralised, filtered, made up to 500 c.c., and titrated by Fehling's solution. The sugar found, multiplied by 0·916, gives the amount of starch. The use of lead acetate for decolorising the solution introduces errors. The best mode of making the titration is to measure out, dilute, and heat the copper solution, then add at once a certain quantity of the sugar solution, boil for two minutes, and examine the upper liquor. If too much or too little of the sugar has been used, a fresh quantity of the copper solution should be taken. The use of potassium ferrocyanide as indicator is altogether misleading.

M. J. S.

Determination of Starch by Baryta. By F. SEYFERT (*Zeit. angewand. Chem.*, **1**, 126—129).—Asboth's method (Abstr., 1887, 868) is capable of giving results agreeing very closely amongst themselves when the conditions are kept precisely similar. A very important point is the strength and quantity of the alcohol used, the limits being comparatively narrow within which the amount of baryta precipitated is such as to correspond with the formula given by Asboth. The use of 50 c.c. of 45 per cent. alcohol in making up the mixture to 250 c.c. gives results nearest the truth. The starch must be very thoroughly gelatinised, one to two hours on the water-bath being necessary. The baryta solution should not contain less than 23 grams of barium oxide in the litre. M. J. S.

New Extraction Apparatus. By C. M. KING (*Chem. News*, **57**, 235—236).—This apparatus consist of a wide test-tube, *a*, with a quill tube *b* fused into the bottom and extending some way both inside and beyond *a*. The end of *b* inside *a* is cut off obliquely, and upon its apex rests the closed end of a slightly wider tube *c*, which reaches nearly to the bottom of *a*. *a* is inclosed in a still wider tube *d*, and is supported on small projections just above the narrower lower end of *d* into which the quill *b* tube extends from the bottom of the test-tube *a*. In operation, the substance to be extracted is placed in the space between the test-tube *a* and the tube *c*, and rests on a plug of glass-wool attached to the tube *c* in such a way as to leave a clear, liquid space below. The upper part of the tube

d is connected with a condenser, whilst the narrow part passes through the cork of the flask containing the solvent; as distillation proceeds, the test-tube *a* and the space between the tubes *b* and *c* gradually fill up until the open end of *b* is reached, when the extract syphons off into the flask below; in this manner the solvent continues to circulate until the operation is complete. D. A. L.

Determination of Fat in Milk. By B. Röse (*Zeit. angewand. Chem.*, **1**, 100—107).—About 20 grams of the milk is mixed with 2 c.c. of ammonia, then 45 c.c. of alcohol, and 120 c.c. of a mixture of equal parts of ether and light petroleum, and is shaken in a stoppered burette of 230 c.c. capacity. The volume of the ethereal layer is read off and 25 c.c. of it is evaporated in a tared flask, the fat being dried by aspirating dried air through the flask for 10 minutes, whilst heating in a glycerol-bath at 90°. An addition of 0·015 per cent. should be made for fat remaining in the aqueous layer.

When milk is evaporated with sand, the fat must be extracted the same day; if the dry residue is kept, the amount of fat obtainable from it diminishes very seriously (see next Abstract). M. J. S.

Determination of Fat in Milk. By H. Schreib (*Zeit. angewand. Chem.*, **1**, 135).—In the course of a considerable experience of the sand process for determining fat in milk, the author has not encountered results confirming the statement of Röse (preceding Abstract), that the dry residue if preserved for several days before extraction with ether yields less fat than if extracted at once. In special experiments made for the purpose, the same amount of fat was obtained by extracting on the fourth day as on the first, and that whether the residue were preserved in paper (as in Röse's experiments) or in the basins in which the evaporation took place.

M. J. S.

Adulteration of Butter. By P. Bockairy (*Bull. Soc. Chim.*, **49**, 247—251, and 331—335).—15 c.c. of the dry, melted and filtered butter are poured into 15 c.c. of toluene, placed in a graduated tube, and 40 c.c. of alcohol (96·7° Gay-Lussac) added; at 18° the toluene, containing the fat in solution, remains at the bottom of the vessel. The tube is then placed in water at 50°, and the mixture agitated, after which it is placed in water at 40° for half an hour. In the case of pure butter no precipitate, or only a slight cloud, is formed; if, however, extraneous fats are present, an oily or flocculent precipitate deposits, from the quantity and character of which the amount and nature of the adulteration can be roughly estimated.

The results obtained with pure butter from various sources, and with the same butter after the addition of varying quantities of fats, are given in tabular form. If 2 to 3 c.c. of precipitate are obtained, the butter is most probably adulterated, but when the precipitate exceeds this volume the sample is certainly impure. 10 per cent. of added oleomargarine, margarine, olive oil, beef-fat, &c., causes a precipitate of from 10 to 12 c.c.; 75 per cent. of the same impurities produce

a precipitate of from 19 to 22 c.c., intermediate quantities give proportional precipitates. F. S. K.

Detection of Cotton-seed Oil in Olive Oil. By F. Jean (*Dingl. polyt. J.*, **268**, 101).—The author has obtained good results with Becchi's method, which requires the following solutions:—(1) 1 gram of silver nitrate dissolved in 100 c.c. each of alcohol and ether, (2) 85 parts of amyl alcohol (boiling at 130—132°), and 15 parts of pure rape oil. 10 c.c. of the oil to be examined is mixed in a glass tube with 11 c.c. of the silver solution and 8 to 10 c.c. of the rape oil solution. The mixture is then heated for 15 minutes in a water-bath. If cotton-seed oil is present, a brown coloration is obtained. D. B.

Detection of Sesame Oil in Cocoa-butter. By P. Zipperer *Chem. Zeit.*, **11**, 1600).—Sesame oil is frequently added in small quantities to chocolate to give it a glossy surface and good fracture. When such chocolate is extracted for analysis, the sesame oil is dissolved along with the cocoa-butter. It may be detected by gently warming 2 c.c. of the suspected cocoa-butter with a freshly prepared mixture consisting of 1 c.c. hydrochloric acid, sp. gr. 1·18, and 0·05—0·1 gram of cane-sugar; the production of a raspberry-red coloration indicates the presence of sesame oil; pure cocoa-butter under similar circumstances gives a yellowish to dark-brown colour. In this manner, an admixture of $\frac{1}{50}$ per cent. of sesame oil may be detected. D. A. L.

Detection of Acetanilide. By D. Vitali (*Chem. Centr.*, 1888, 424, from *L'Orosi*, **11**, 1—5). Acetanilide (antifebrin) could not be detected in the urine of people who had consumed this compound. The following method was used: the urine, either in its original state or neutralised with ammonia, was extracted with five times its volume of ether. The ethereal solution gave no residue. Acetanilide gives the following colour reactions. A trace of it gives a blue coloration with a few drops of a solution of bleaching powder and a crystal of phenol. Mixed with a solution of potassium chlorate in sulphuric acid, it produces a red colour, which is changed to yellow by water, by heating to blood-red. With a crystal of potassium nitrite and a drop of concentrated hydrochloric acid, acetanilide produces a yellow colour, which, on heating, changes through green to blue. On evaporation to dryness, an orange-coloured residue remains, which is coloured red by ammonia. J. W. L.

Detection of Pyridine Bases. By Weppen and Lüders (*Chem. Zeit.*, **12**, 151).—Cadmium chloride has been employed for the detection of pyridine in denaturated spirit; a 5 per cent. solution of the salt should produce a decided turbidity in a few seconds, in an aqueous solutions of 1 of base in 250 of water. Many operators have failed to obtain this reaction, and the authors now show that this is very probably due to variations in the composition of the cadmium chloride. In two samples of this salt, one giving the reaction contained more

cadmium than the other which did not produce the turbidity. The test is uncertain therefore. D. A. L.

Estimation of Morphine in Opium. By E. F. Teschemacher and J. D. Smith (*Chem. News*, **57**, 244—245).—A controversial communication referring to R. William's paper (*loc. cit.*, 134—135, and this vol., p. 635). Incidentally it is noted that all they "pretend to do" by their method "is to return results certifying to the amount of pure crystalline morphine which can be obtained from a given sample of opium," and moreover they assert that they obtain more morphine than any other analysts do; but do not place implicit reliance on their method, nor do they believe in its giving accurate results. D. A. L.

Delicate Test for Morphine. By J. L. Armitage (*Phil. J. Trans.*, **18**, 761).—When ferric chloride is added to a solution of a morphine salt, a bluish-green coloration (imperceptible in dilute solutions) occurs due to the reduction of some of the ferric chloride to the ferrous state, and when potassium ferricyanide is added, a deep-blue coloration takes place, or in very dilute solutions, a greenish-blue coloration. One part of morphine salt in 100,000 will give this reaction unmistakably after standing a few minutes. This is a delicate confimatory test in the absence of other reducing substances which would give the same reaction, but several other alkaloïds submitted to this test failed to give the coloration. R. R.

Difference between the Colouring Matters of Bilberry and Wine: Spectroscopic Wine Testing. By H. W. Vogel (*Ber.*, **21**, 1746—1753).—The fresh colouring matters of bilberries and wine are similar but not identical. They are readily distinguished from one another by their absorption-spectra after they have been carefully neutralised with ammonia, or better still when they are treated with a trace of a solution of alum before neutralising with ammonia. The unsuccessful results obtained by others are attributed to the use of too concentrated wines and the addition of too much ammonia. When fermented or when kept long, the colouring matters can no longer be distinguished from each other. The positions of the absorption-bands in each case are shown in a table. (Compare Andree, *Arch. Pharm.*, **13**, 90.) N. H. M.

Testing for Santonin. By L. Knapp (*Dingl. polyt. J.*, **268**, 42—44).—In the process of extraction and purification of santonin, solutions and extracts are obtained which may contain santonin and therefore require to be tested for the presence of this substance. Linde's reaction consists in mixing a few drops of the solution with concentrated sulphuric acid, whereby a violet colour is obtained varying in depth of tint with the amount of santonin present in the solution. This test can, however, be applied only to colourless solutions. In order to examine coloured solutions, Kossakoffsky recommends precipitating the colouring matter with lead acetate and then treating the filtrate with sulphuric acid. D. B.

The Vienna Gravimetric Method of Estimating Tannin in Concentrated Solutions.—By R. Koch (*Dingl. polyt. J.*, **268**, 329—334).—The author refers to the publication of the results of some analyses made by Simand, which show that the amount of extract and the percentage of tannin are not influenced by the quantity of bark operated on. The same chemist also asserts that with extracts of different concentrations the same amounts of soluble and insoluble tannin are obtained. As these conclusions are in opposition to Councler's results and disagree with the author's experience of the Vienna method, the following experiments were carried out:—

60 grams of fir bark was extracted by means of the Vienna extraction apparatus and the concentrated solution diluted to 1 litre. It was then again heated in order to redissolve any precipitated tannin, after which it was allowed to cool to 18°.

Experiment 1.—50 c.c. of this solution was evaporated to dryness on a water-bath. The residue weighed 0·870 gram, equal to 29 per cent.

Experiment 2.—50 c.c. was diluted to 150 c.c., warmed, allowed to cool to 18°, filtered, and 50 c.c. of the filtrate evaporated to dryness and weighed. The residue gave 0·274 gram = 27·4 per cent.

Experiment 3.—50 c.c. of the concentrated solution yielded on evaporation 0·736 gram of residue, corresponding to 24·52 per cent. of solid matter.

Experiment 4.—50 c.c. of the concentrated solution was filtered, diluted with three times the volume of water, and 50 c.c. of this evaporated to dryness. The residue weighed 0·245 gram = 24·5 per cent.

From these results the author draws the conclusion that different amounts of soluble tannin are obtained, when 20 or 60 grams of bark are extracted with one litre of water. Similar results were obtained with extracts of quebracho bark subjected to the above treatment in a solid and pasty form. The author recommends therefore for the estimation of the soluble tannin, the use of a small quantity of bark and a considerable amount of water, whilst for the solids other than tannin it is best to employ for extraction a large quantity of bark. D. B.

Estimation of Tannin. By C. Collin and L. Benoist (*Chem. News*, **57**, 214).—In the process recommended, the following solutions and apparatus are required. A normal solution of tannin containing 5 grams of pure dry tannin and 0·5 c.c. of a solution of mercuric iodide (1 in 10, the iodide being dissolved by means of an equal weight of iodide of potassium) in a litre of water. A standard solution of gelatin, containing 5 grams of gelatin dissolved in a litre of water; it is clarified with egg albumin, mixed with 0·5 c.c. of the mercuric iodide solution and made slightly alkaline with soda. A calcium acetate solution containing 50 grams of the salt dissolved in a litre of water, which is filtered and a few drops of mercuric iodide solution are added. The mercuric iodide is intended to prevent fermentation or fungoid growths. Besides these a 1 per cent. solution of methylene-blue, a 4 per cent. solution of Nicolson blue, and a 1 per cent. solution of black blue, "N.B.I.," for use with coloured tannins and

extracts must be at hand. The apparatus consists of a burette to read to $\frac{1}{100}$ths of c.c. delivering 4 drops to a tenth c.c., and a stoppered test-tube 3 cm. in diameter, and marked at 60 c.c. The gelatin is standardised by taking 1 c.c. of the solution in the test-tube, adding two drops of methylene-blue and 5 c.c. of calcium acetate solution, filling up to the mark with water at 75° to 80°. The quantity of tannin required to precipitate this gelatin is then ascertained by running in normal tannin solution a little at a time, agitating after each addition, until the solution is colourless. The quantity of tannin in any solution may then be determined by employing it instead of the normal solution for the precipitation of the gelatin. Strong tannin solutions should be diluted before titration. D. A. L.

Spectroscopic Examination of Blood. By G. Linossier (*Bull. Soc. Chim.*, **49**, 691—694).—Contrary to the opinion almost universally held, it is not the absorption-bands of oxyhæmoglobin which give the most sensitive spectroscopic reaction, but those of reduced hæmatin (the hæmochromogen of Hoppe-Seyler). The author recommends the following method for the spectroscopic examination of blood:—The blood-stain is dissolved in water with the usual precautions and examined for the spectrum of oxyhæmoglobin. A drop of a freshly prepared solution of sodium hyposulphite is then added; the spectrum of reduced hæmoglobin instantly appears. Finally, one or two drops of a concentrated solution of soda are added; the hæmoglobin is thus decomposed into globulin and reduced hæmatin, the spectrum of which consists of two bands. The more intense band is the only one which can be distinctly observed when the solution of blood is very dilute; it is situated at an equal distance from the D and E lines, and occupies the luminous region which separates the two bands of oxyhæmoglobin; its mean position corresponds with that of the ray whose wave-length is $\lambda = 557$. The second band is more diffuse, and is situated between the lines E and B, its mean position corresponds with $\lambda = 522$. A slight elevation of temperature favours the appearance of this spectrum which, if the solution is very dilute, may only manifest itself after the liquid has been completely cooled.

The last experiment gives positive results only when the solution of blood is extremely dilute, and it is advisable, therefore, to make sure that the spectrum observed is really that of hæmatin. This can be done by the following tests:—The band ought to disappear if the liquid is heated to 50° without agitating, and reappear on cooling. The band ought to disappear when the solution is agitated in the air, and reappear when a drop of sodium hyposulphite is added. If the colouring matter has been radically changed by putrefaction, the spectroscopic reaction of reduced hæmatin may be the only test by which the presence of blood can be proved. In some cases, the colouring matter has become insoluble in water; the stain is then dissolved in tolerably strong ammonia, and the oxyhæmatin is best reduced by one or two drops of a solution of ferrous sulphate and tartaric acid. The spectrum of such a solution shows clearly the first band of the reduced hæmatin.

By this method of spectroscopic examination, blood can often be detected in cases where crystals of hæmin cannot be obtained.

F. S. K.

Detection of Blood Stains. By FERRY DE LA BELLONE (*J. Pharm.* [5], **17**, 253—255, from *Rép. de Pharm.*).—If the stains are on linen or cloth, some filaments of the fabric are placed on the surface of a solution of sodium chloride, 1 : 1000, in a small glass tube; after some hours the liquid takes a brownish-rose tint. If spectroscopic examination of the liquid indicates hæmoglobin, the search for blood corpuscles becomes important. A drop or two of concentrated chloral solution is added to the liquid, when a rose-coloured precipitate is quickly deposited. The supernatant liquid is removed by means of a pipette, a drop of the deposit is spread out on a glass slide, and carefully warmed over a lamp; a reddish coagulum is formed, and a clear liquid which is removed by filter-paper. The coagulum is stained with a little magenta, washed, and to it is added a drop of dilute acetic acid. The globules stained a bright-red are then visible under the microscope. When the stain is found on instruments, wood or stone, &c., the powder obtained by scraping is placed in a fine bag and suspended in the salt solution. When the powder is mixed with earth, the clay usually present causes a rapid precipitation in the salt solution; in this case, the reddish particles should be picked out, under the microscope, for testing. In all cases, the results may be confirmed by the formation of hæmin crystals. For this purpose, one or more drops of the brown-rose solution is taken before the addition of chloral, and carefully evaporated to dryness on a slide. It is then covered and warmed with a drop of glacial acetic acid when the crystals of hæmin appear in large numbers on cooling.

J. T.

Detection of Albumin, Propeptone, and Peptone. By C. POSNER (*Zeit. anal. Chem.*, **27**, 408—409, from *Arch. Physiol.*, 1887, 495).—The liquid (urine, &c.), is rendered alkaline and a solution of copper sulphate so dilute as to be nearly colourless is poured on its surface. Peptone in the cold, or albumin after heating, gives a reddish-violet ring at the point of contact.

M. J. S.

General and Physical Chemistry.

Relations between the Composition and the Absorption-spectrum of Organic Compounds. By G. KRÜSS (*Zeit. physikal. Chem.*, **2**, 312—337).—The absorption-spectra of solutions of derivatives of indigo, fluoresceïn, and anthracene were examined, the solvents being chloroform or concentrated sulphuric acid for the indigo and anthracene, and alcohol or water for the fluoresceïn-derivatives. The wave-lengths of the lines of maximum absorption are given for every compound, the number of compounds examined being in all 64. From these observations, and those of others, the author finds generally that the substitution of a methyl-, ethyl-, methoxyl-, or carboxyl-group, or of bromine for hydrogen, moves the absorption lines further towards the red, and that the substitution of a nitro- or amido-group causes a movement further towards the violet. The only exceptions found were for dibromamido-indigo and bromalizarin in the first case, and for the alcoholic solutions of tetranitro- and dibromodinitro-fluoresceïn in the second, the behaviour of these being directly opposite to the normal one. The aqueous solutions of the last two compounds follow the general rule. The displacement caused by the introduction of any of the above groups appears to depend in amount on the nature of the compound into which the group enters, but general rules for this cannot be deduced.

If the production of absorption lines be looked on as caused by absorption, by a compound, of those ether waves which are capable of vibrating in unison with its own molecules, then the number of vibrations of such molecules would be given by $n = v/\lambda$, v being the velocity of light, λ the wave-length corresponding with the absorption line. Increase of λ by movement of the line towards the red would mean a decrease in n, and decrease of λ by movement towards the violet, an increase of n. From this it would appear that the introduction of methyl, ethyl, methoxyl, carboxyl, or bromine into a molecule, in place of hydrogen, retards; the introduction of a nitro- or amido-group, accelerates its vibrations. H. C.

Cause of the Change of Specific Rotatory Power under the Influence of Various Solvents. By G. J. W. BREMER (*Rec. Trav. Chim.*, **6**, 255—261).—A reply to Přibram (Abstr., 1887, 755), in which the author endeavours to show that Přibram's results do not prove conclusively that the specific rotatory power of a solution continually decreases as it is more and more dilute. F. S. K.

Reactions in Secondary Coils (Elements). By LODSINSKY (*J. Russ. Chem. Soc.*, 1887, 542—548).—The author recapitulates the results obtained by Gladstone and Tribe, Frankland, Aron, Tcheltsoff, Beklemisheff, and Kanin, and shows that it is necessary to investigate

exactly the dependence of charge, quantity of electricity, and E.M.F. on the concentration of the sulphuric acid used. It is first shown by quantitative experiments that the reaction really consists in the formation of a sulphate: $Pb + PbO_2 + 2H_2SO_4,aq = 2PbSO_4 + 2H_2O + aq$. The reverse action is shown to take place on charging the elements, so that Aron's hypothesis that hydrated peroxide of lead is formed is erroneous. It is shown further that Frankland is right in recommending to charge the batteries in intervals, for time must be given for the exchange by diffusion of the weaker acid formed inside the plates with the stronger acid outside, otherwise formation of PbO alone, but no sulphate, would take place, and the energy of the reaction $PbO + H_2SO_4,aq$ would be lost. From the absence of sulphuric acid in the plates, the formation of PbO_2 is explained, as otherwise the PbO first formed would become converted at once into the sulphate. It is shown by quantitative experiments that owing to slow diffusion, the concentration of the acid becomes greater than it was at the commencement of the experiment, both on discharging as well as on charging. B. B.

Theory of the Dissociation of Electrolytes. By W. Ostwald (*Zeit. physikal. Chem.*, **2**, 270—284).—Various considerations are advanced in support of Arrhenius' theory of the dissociation of electrolytes. The difficulty in recognising dissociation among salts which are looked on as chemically very stable, lies in the confusion of the affinity with which the elements are combined in such compounds, with that which appears active in the behaviour of these compounds towards others. The two are not of the same but of opposite properties. The more energetic a substance is chemically, the easier will it be split up into its atoms; and the greater the power with which its elements are held together, the less will be its chemical activity. The existence of atoms of such elements as potassium and sodium in water can be understood when it is remembered that their condition as ions with large electric charges is very different from that of the same atoms in the so-called free state.

The author has shown, from the action of acids, that certain so-called coefficients of affinity may be deduced for these, which are independent of the nature of the chemical reaction from which the coefficients are obtained, and are proportional to the electrical conductivities. These affinity coefficients would, in the light of the present theory, represent nothing more than a measure of the dissociation of the acids. For the greater the extent to which dissociation into hydrogen and free ion had already taken place, the more readily would the combination of either of these two with other groups take place. And since the dissociation is independent in amount of the nature of the reaction into which the acid enters, this would explain how for the same acid the same coefficient of affinity is obtained in every reaction.

The equation $(\mu_\infty - \mu_v)\mu_\infty v/\mu_v^2 = c$, which the author has deduced from the dissociation theory (this vol., p. 1020), expresses the whole electrical behaviour of binary electrolytes. Thus as v increases, μ_v must also increase until at length it reaches the limiting value μ_∞,

which is the law of the increase of the molecular conductivity with increasing dilution. μ_∞ is the conductivity of the wholly dissociated electrolyte, and since in this the motion of each ion is independent of that of the other, μ_∞ should be deduced simply from the sum of these motions, and as a matter of fact μ_∞ may be represented as the sum of two numbers, one of which depends on the nature of the negative and the other on that of the positive ion. This is only true for infinite dilution, for with greater concentration the law is merely approximate, owing to the dissociation not being complete. With acids and bases of low conductivity, μ_v is small in comparison with μ_∞, and $\mu_\infty - \mu_v$ becomes nearly constant, so that $\mu_v^2 = v$ const. This is the law already established, that where the conductivity is low, it increases with rising dilution in proportion to the square root of the volume. If the dilutions $v_1, v_2 \ldots$ be measured at which electrolytes have the same relative conductivity, that is where the relation between μ_v and μ_∞ is the same for all, then $\mu_\infty(\mu_\infty - \mu_v)/\mu_v^2$ will be constant, and $v_1/c_1 = v_2/c_2 = v_3/c_3 = \ldots$, or the above dilutions stand in a constant relation, depending only on the nature of the electrolytes. The increase in the conductivity of all monobasic acids or monacid bases follows then the same law, which has been already established.

If $m = \mu_v/\mu_\infty$, we have $m^2/(1 - m)v = C$. This relation has been tested with various substances, and found to hold good with remarkable accuracy for both weak and strong acids from $m = 0{\cdot}7$ to $m = 76$. Above this limit, the experimental errors have too great an influence to allow of its being tested. The effect of increasing the concentration of the solution also is to decrease the constant slightly. This is owing to the change in the molecular volume and in the viscosity, and if corrections for both be applied, the constancy becomes more apparent, even up to 10 per cent. concentrations. H. C.

Raoult's Law of Freezing. By W. Hentschel (*Zeit. physikal. Chem.*, **2**, 306—311).—The author describes a new form of apparatus for the determination of the freezing point. Experiments were made with various substances in benzene and acetic acid solutions, which tend on the whole to confirm the truth of the law. The numbers for acetic and formic acids in benzene show that they are present as double molecules, but benzene in acetic acid gives the normal value for a single molecule. The exception shown by acetic and formic acids leads the author to doubt the general validity of Raoult's law. The substance prepared by passing chlorine through a mixture of methyl formate and chloroformate (*J. pr. Chem.*, **36**, 468; Abstr., 1888, 249) gave a molecular weight $\frac{1}{2}C_4H_3Cl_5O_4$, and from this and former investigations would appear to be a mixture of chloromethyl and dichloromethyl chloroformate in molecular proportion.

H. C.

Theory of Liquids. By G. P. Grimaldi (*Zeit. physikal. Chem.*, **2**, 374—377).—In criticism of Konowaloff's paper (this vol., p. 1019), the author shows that his own equation for the expansion $v_t = v_0/(1 - k_1t - k_2t^2)$ gives values for the coefficients of expansion which bring the calculated numbers for ϕ in the case of ether and

pentane into a closer agreement with the observed values than was obtained by Konowaloff. H. C.

Hypothesis of the Dissociation of Salts in very Dilute Solutions. By M. PLANCK (*Zeit. physikal. Chem.*, **2**, 343).—In answer to Wiedemann (this vol., p. 1021), the author points out that the value of E in the equation $E = \text{const. } n_1/n$ is not altered by a change in n. For the constant has the value θ^2/q, where θ is the temperature and q the latent heat of freezing; q combines in the denominator with n to the product nq, and this number, which is the latent heat of fusion of the whole solvent, depends directly on the weight and not on the size of the molecules of the latter. The author has further shown (*Ann. Phys. Chem.* [2], **34**, 149) that equilibrium in a solution of infinite dilution is independent of any polymerisation of the solvent, although not absolutely so in moderately dilute solutions. H. C.

Theory of Isohydric Solutions. By S. ARRHENIUS (*Zeit. physikal. Chem.*, **2**, 284—295).—An extension of the author's dissociation theory. Two solutions are isohydric, the electrical conductivities, or in other words the electrolytic dissociation of which remains unchanged when they are mixed. Let there be two acids, H_mA and H_nB, dissolved in water so that the volumes of the solutions are V_a and V_b. Further, let a molecules of the first remain undissociated, and α molecules be dissociated into the ions mH and A, and let b and β be the corresponding numbers for the second. Then if the solutions are isohydric, from considerations based on Van't Hoff's assumption of the analogy of the condition of salts in dilute solutions to the same in the gaseous state, we get $(m\alpha + n\beta)/(V_a + V_b) = m\alpha/V_a = n\beta/V_b$. These relations give expression to conditions which the author has already shown to exist in isohydric solutions (*Ann. Phys. Chem.* [2], **30**, 54; Abstr., 1887, 415).

The theory thus formulated is applied to the case of the dissociation of ammonia and ammonium chloride, when both are present in the same solution, in order to interpret the influence which ammonium salts have in retarding the saponification of ethyl acetate by ammonia, and which is in connection with the reduction of the conductivity of ammonia by ammonium salts. The theory is also extended so as to apply to the case of any number of electrolytes in solution, and leads to results similar to those which have been obtained from reasoning based on thermodynamical principles. The author concludes that the properties of dilute solutions generally, may be deduced from Van't Hoff's analogy between the gaseous state and the state of solution and the hypothesis of the dissociation of electrolytes. H. C.

Experiments on the Diffusion of Aqueous Solutions. By J. D. R. SCHEFFER (*Zeit. physikal. Chem.*, **2**, 390—404).—The solutions were allowed to diffuse in a cylindrical vessel, the diffusate being divided into four equal layers, and the amount of dissolved substance in each determined. By means of Stefan's tables, the coefficient of diffusion k of each solution was then calculated. For HCl,nOH_2 the value of the coefficient rises with the concentration from $n = 10$ to

$n = 4{\cdot}5$, k being for the first, at 0° about 1·8, and for the second about 2·4. There is a rise with the concentration, for the weaker solutions also, but it is not so marked. Concentrated hydrochloric acid solutions diffuse more rapidly into solutions of weak hydrochloric acid than into water. This is explained on the hypothesis that concentrated solutions contain molecular aggregates of hydrochloric acid with relatively small quantities of water, and that on dilution, molecular aggregates richer in water are formed, this action being the more complete the greater the dilution. The results further show that the coefficient of diffusion of nitrates and some other salts, such as sodium thiosulphate and magnesium sulphate, decreases with rising concentration of the solution, whereas for nitric and sulphuric acids and calcium chloride it increases as with hydrochloric acid, although not to anything like the same extent. The author looks on this change in the value of the coefficient with changing dilution as due to molecular actions between the dissolved substance and the water. The fact that the coefficient becomes more nearly constant when a concentrated solution diffuses into a weaker solution of the same salt instead of into water, is given in support of this, since it may be assumed that in the former case the molecular aggregates have not to undergo such far reaching changes as in the latter. The coefficients also approximate to constants as the dilution of the solutions examined increases, which may be traced to the same cause. Generally, then, the coefficients of diffusion of concentrated solutions are by no means constant, and for purposes of comparison with other properties, the coefficients of dilute solutions can alone be employed. H. C.

Vapour-tensions of Ethereal Solutions. By F. M. Raoult (*Zeit. physikal. Chem.*, **2**, 353—373).—For solutions in ether of substances whose vapour-tension is very small in comparison with that of the ether, the law holds $100\,f'/f = 100 - kN$, where f is the vapour-tension of ether and f' that of the solution at the temperature of the experiment, N the number of molecules of substance in 100 mols. of the solution, and k a constant depending on the nature of the dissolved substance. The substances examined, with the values found for k, are turpentine 0·90, aniline 0·90, ethyl benzoate 0·90, methyl salicylate 0·82, and nitrobenzene 0·70. It will be seen that the values of k do not alter greatly for different substances. For dilute solutions in which N is not greater than 15, the value of k for all substances may be taken as unity. This does not hold for N less than 2, in which case the law appears to be more complicated, but this might be due to the increased difficulty of experimenting. If instead of the number of molecules of the dissolved substance, the number of molecules of ether in 100 mols. of the solution $N' = 100 - N$ be taken, and the value of k is unity, the law takes the simple form $100\,f'/f = N'$, or in other words, the tension of the ether vapour of dilute ethereal solutions depends simply on the number of ether molecules in 100 mols. of the solution, and is quite independent of the nature of the dissolved substance. For concentrated solutions in which N is greater than 70, small variations

from the law which occur, may probably be due to the increase in the difficulties and errors of experiment. The law is independent of the temperature between the limits 0° and 20°.

The relation $100\,f'/f = 100 - kN$ may also take the form $(f - f')/f = kN/100$. Calling $(f - f')/f$ the relative decrease in the vapour-tension, we find then that for all ethereal solutions this number is simply proportional to the number of molecules of the substance dissolved in 100 mols. of the solution. Since for $N < 15$ we get $k = 1$, and therefore $(f - f')/fN = 0{\cdot}01$, we find also that the relative decrease in the vapour-tension of a dilute ethereal solution, divided by the number of molecules of non-volatile substance in 100 mols. of the solution, is equal to the constant quantity 0·01. This relation was tested with a number of substances differing widely in chemical composition, and found to hold good within experimental error. It may be used for determining the molecular weights of substances soluble in ether, for if the weight p of a substance of molecular weight M be dissolved in 100 grams of ether, then $M = 0{\cdot}74\,f'p/(f - f')$. The author does not, however, recommend the method, both on account of the experimental difficulties and the fact that it can only be used for substances which boil above 150°; as a means of determining molecular weights, therefore, it cannot be compared with the cryoscopic method. H. C.

Vapour-tension of Dilute Solutions of Volatile Substances. By M. Planck (*Zeit. physikal. Chem.*, **2**, 405—414).—If n and n_1 be the number of molecules of the solvent and the dissolved substance in a dilute solution, the vapour of which contains n' and n_1' of each, then the concentration of the dissolved substance in the solution is given by $c_1 = n_1/(n + n_1)$, and in the vapour by $c_1' = n_1'/(n' + n_1')$, and from the conditions of equilibrium $c_1 - c_1' = \log K$ and $\log c_1'/c_1 = \log K_1$, where K and K_1 are both functions of the temperature θ and the pressure p. The values of the functions are unknown, but the differential coefficients may be calculated, and are for both K and K_1 of the forms $\delta(\log K)/\delta\theta = Q/\theta^2$ and $\delta(\log K)/\delta p = -V/\theta$, Q being the amount of heat, measured mechanically, which is given to the system at constant temperature θ and pressure p, in order to establish the above conditions of equilibrium, and V the attendant alteration in volume.

For the equation $c_1 - c_1' = \log K$, the value of K may be calculated in terms either of p or of θ, by introducing the vapour-tension p_0 of the pure solvent at the temperature θ, or the boiling point θ_0 of the pure solvent at the pressure p, and expanding in powers of $(p - p_0)$ or $(\theta - \theta_0)$. The first gives $c_1 - c_1' = (p_0 - p)/p_0$, the second $c_1 - c_1' = (\theta - \theta_0)Q/\theta_0^2$. The first of these relations expresses in words that the relative decrease in the vapour-tension is equal to the difference in the concentration of the dissolved substance in the liquid and in the vapour. The author has calculated the values which it gives for c_1' for the two cases of formic acid in water, where $p_0 > p$, and for isobutyl alcohol in water, where $p_0 < p$, from observations of the vapour-tension by Konowaloff. It is obvious that according as p is greater or less than p_0, that the amount of the dissolved substance in

the liquid is less or greater than in the vapour, and that the concentration will be the same in both when $p = p_0$, a result which has already been enunciated by Konowaloff (*Ann. Phys. Chem.* [2], **14**, 48). For non-volatile substances $c_1' = 0$, and we get the well-known law of the molecular reduction of the vapour-tension; for insoluble gases $c_1 = 0$ and $c_1' = (p - p_0)/p$, where $p - p_0$ is the pressure exercised by the gas, and p_0 that of the solvent in the mixed vapour, the equation shows that the pressures are proportional to the concentrations, since that of the solvent is very nearly unity, which is Dalton's law. The second relation $c' - c_1' = (\theta - \theta_0)Q/\theta_0^2$ expresses, if Q be taken for simplicity as the heat of vaporisation of 1 mol. of the solvent, that the difference in the concentration of the dissolved substance in the liquid and in the vapour is equal to this quantity multiplied by the rise and divided by the square of the boiling point. The values of c_1' have been calculated from this relation, also for aqueous solutions of formic acid and isobutyl alcohol, and are found to agree very well with those calculated by the first.

From the equation $\log c_1'/c_1 = \log K_1$ the value of c_1' may be obtained by substituting the known value of $d \log K_1$ for $d\theta$ and dp, but the expression contains Q and V, the relations of which to one another are unknown, and it cannot therefore be used practically.

All the above relations are only applicable on the assumption that the molecules of the dissolved substance are in their normal condition, and are neither dissociated nor combined one with another. They also only apply in the case of dilute solutions, but within these limits they include the most extreme cases, both those in which the dissolved substance is non-volatile and those in which it is absolutely volatile as for insoluble gases. H. C.

A Criticism on "The Arrangement in Space of the Atoms in the Molecules of Organic Compounds." By A. Michael (*J. pr. Chem.* [2], **38**, 6—39).—The author argues that the agreement between experiment and theory in the phenomena studied by Wislicenus and explained by his extension of Van't Hoff's hypothesis (*Abhandlung. der K. Sächs. Gesell. der Wiss.*, 1887) is only apparent, and is based partly on unjustifiable assumptions, partly on one-sided explanations, and partly on the omission of a large number of facts which would lead to exactly contrary conclusions; and shows that for many substances as many as three different configurations are equally probable. F. S. K.

The Different Forms of Heterogeneous Chemical Equilibrium. By H. W. B. Roozeboom (*Rec. Trav. Chim.*, **6**, 262—303; compare Roozeboom, *Rec. Trav. Chim.*, **5**, 408).—The term "transition point" should be reserved for single points in heterogeneous equilibria, which present the greatest analogy with triple points in physical equilibria; such points are not confined exclusively to the equilibrium of condensed systems. As the transition point varies with the pressure, in order to render Van't Hoff's definition (*Zeit. physikal. Chem.*, **1**, 166) correct it would be necessary to add "at constant pressure," or "at the pressure of one atmosphere." In the latter case, transition points

which only exist under a higher pressure (compare Roozeboom, *Rec. Trav. Chim.*, **5**, 396) would be excluded, and in the former case the definition would include the whole series of temperatures at which equilibrium between liquid and solid systems is possible, but would at the same time lose all that is characteristic of the transition point.

The distinctive feature in the equilibrium of condensed systems is the absence of gaseous compounds, but such equilibria belong, nevertheless, to the category of heterogeneous equilibrium. The equilibrium of condensed systems presents a special form which is best compared to the physical equilibrium between the solid and the liquid state of the same substance, and amongst the examples of heterogeneous equilibrium those which are analogous to the volatilisation of solids can be distinguished from those which are more comparable to the evaporation of liquids.

Equilibria differ also in the degree of heterogeneousness. In equilibria of systems formed of a single substance, no distinction is necessary, but in those formed of two or more substances, the degree of heterogeneousness is judged best by a comparison of the number of constituent substances which enter into the system with the number of coexistent phases, that is to say, with the different heterogeneous complexes into which one or more of the substances of the system enter, either in definite combination or in variable proportions. These phases may differ in state (solid, liquid, gaseous) and in composition, as far as it concerns liquid and solid states in which two or more complexes may coexist unmixed.

Gibbs has shown (*Trans. Connect. Acad.*, **3**, 152) that a perfectly definite state of equilibrium exists when there are $n + 1$ phases of n substances. The coexistence of $n + 1$ phases is, therefore, the necessary and sufficient condition for having at a constant temperature a state of equilibrium in which the pressure and the composition of the liquid and gaseous phases remain constant independently of the volume. The author names this state complete heterogeneous equilibrium. There is incomplete heterogeneous equilibrium when there are n substances in less than $n + 1$ phases. Complete becomes, therefore, incomplete heterogeneous equilibrium when one or more of the phases are suppressed or disappear owing to a change of temperature or pressure; as the number of phases decreases, heterogeneous equilibrium becomes more and more incomplete, until homogeneous equilibrium is established.

The author classifies the phenomena of dissociation according to the physical state of the coexistent phases, into three divisions (I, II, III), which are comparable to one of the three forms of physical heterogeneous equilibrium, and can be expressed by the symbols solid $\rightleftarrows$ vapour, liquid $\rightleftarrows$ vapour, solid $\rightleftarrows$ liquid. Each class is then subdivided (1, 2, 3, &c.) according to the number of substances which enter into equilibrium, and again subdivided (A, B, C, &c.) according to the degree of heterogeneousness as indicated by the number of coexistent phases.

I. *Volatilisation and Analogous Chemical Equilibria.*—(1.) Systems formed of a single substance. Examples:—The isomeric and polymeric transformations of substances which can exist in the gaseous

state, for instance, paracyanogen, amorphous phosphorus. (2.) Systems formed of two substances. (A.) Incomplete equilibrium between one solid and one gaseous phase. Examples:—The absorption of gas by solids (charcoal and certain metals), or the absorption of hydrogen by metallic hydrides (Pd_2H,K_2H). The decomposition of ammonium carbamate, phosphorus pentachloride, mercuric oxide, &c. The phenomena studied by Troost and Hautefeuille (*Ann. Chim. Phys.* [5], **7**, 452), and by Ditte (*Ann. Scient. École Norm.* [2], **1**, 293), of the volatilisation of a simple substance into a gas, with which it can enter into chemical combination, for instance:—

$$2Si_2Cl_6\ (gas) \rightleftarrows 3SiCl_4\ (gas) + Si\ (solid),$$
$$H_2Se\ (gas) \rightleftarrows H_2\ (gas) + Se\ (solid).$$

(B.) Complete equilibrium between two solid and one gaseous phase. Examples:—The decomposition of solids into one solid and one gaseous constituent—

$$IrO_2 \rightleftarrows Ir + O_2 \qquad Pd_2H \rightleftarrows Pd_2Hx + H_{1-x}$$
$$Na_2HPO_4,12H_2O \rightleftarrows Na_2HPO_4,7H_2O + 5H_2O\ (gas)$$
$$SO_2,7H_2O \rightleftarrows 7H_2O + SO_2 \qquad CaCO_3 \rightleftarrows CaO + CO_2.$$

(3.) Systems formed of more than two substances. (A.) Incomplete equilibrium between one gaseous and one solid phase. Example:—

$$NH_4HCO_3 \rightleftarrows \underbrace{NH_3 + CO_2 + H_2O}_{\text{gas.}}$$

(B.) Incomplete equilibrium between one gaseous and two solid phases. Example:—

$$2NaHCO_3 \rightleftarrows Na_2CO_3 + \underbrace{H_2O + CO_2}_{\text{gas.}}$$

(C.) Complete equilibrium between one gaseous and three solid phases. Example:—

$$Na_2SO_4,10H_2O + MgSO_4,7H_2O \rightleftarrows Na_2Mg(SO_4)_2,4H_2O + 13H_2O\ (vapour).$$

II. *Evaporation and Analogous Chemical Equilibria.*—(1.) Systems formed of a single substance. Example:—A liquid and its vapour. (2.) Systems formed of two substances. (A.) Incomplete equilibrium between one gaseous and one liquid phase. Examples:—The solution of a non-volatile substance in a liquid. A liquid and its vapour, the gas enclosing the two constituents at least partially uncombined. The decomposition of a gas into one liquid and one gaseous substance, for instance—

$$2Si_2Cl_6\ (gas) \rightleftarrows 3SiCl_4\ (gas) + Si\ (liquid)\ \text{above } 1000°$$
$$H_2Se\ (gas) \rightleftarrows H_2\ (gas) + Se\ (liquid) \quad ,, \quad 270°.$$

(B.) Complete equilibrium between two liquid and one gaseous phase. Examples:—Physical equilibrium between two only partially miscible liquids and their vapours. (C.) Complete equilibrium between one liquid, one solid, and one gaseous phase. Examples:—Equilibria into

which the hydrates of a gas, the compounds of ammonium salts with ammonia, and the compounds of selenious acid with hydrochloric and with hydrobromic acid enter in the solid state (compare *Rec. Trav. Chim.*, **4**, 361, and **5**, 387). Equilibrium between a salt, its solution, and the vapour of the solvent. (3.) Systems formed of more than two substances. (A.) Complete equilibrium between three liquids and their vapours. Examples unknown. (B.) Complete equilibrium between one gaseous, two liquid, and one solid phase. Examples:—The equilibria studied by Forcrand (*Ann. Chim. Phys.* [5], **28**, 9), in which the solid is a compound consisting of water, hydrogen sulphide, and some chloro-, bromo-, or iodo-derivative of an organic fatty compound, and constituted after the type $CHCl_3,2H_2S,23H_2O$. (C.) Complete equilibrium between two solid, one liquid, and one gaseous phase. Example:—Two salts in their saturated aqueous solutions. (D.) Incomplete equilibrium between two liquid and one gaseous phase. Examples unknown. (E.) Incomplete equilibrium between one solid, one liquid, and one gaseous phase. Example:—A solution of two salts, of which one only is in excess. Solutions of alkaline bicarbonates, double salts, acid salts, and basic salts, which are decomposed by water. (F). Incomplete equilibrium between one liquid phase and one gaseous phase comprising three substances. Examples unknown.

III. *Fusion and Analogous Chemical Equilibria.*—(1). Systems formed of a single substance. Example:—Equilibrium between the two modifications of one substance. (2.) Systems formed of two substances. (A.) Complete equilibrium between one solid and two liquid phases. Examples:—

$$SO_2,7H_2O \rightleftarrows SO_2 \approx\approx x H_2O + H_2O \approx\approx y SO_2$$
$$HCl,H_2O \rightleftarrows HCl \approx\approx x H_2O + H_2O \approx\approx y HCl,$$

(the sign $\approx\approx$ signifies the state of solution). (B.) Complete equilibrium between one liquid and two solid phases. Examples:—Ice and a salt, which exist together in presence of a solution of a salt at the cryohydric point under the pressure of one atmosphere, but which can also exist at a lower temperature if the pressure is increased, for instance—

$$ZnSO_4,7H_2O + 13H_2O\,(\text{solid}) \rightleftarrows 20H_2O \,\ldots\ldots\, ZnSO_4.$$

Also equilibria such as—

$$SO_2,7H_2O + H_2O\,(\text{solid}) \rightleftarrows H_2O \approx\approx x SO_2$$
$$Cl_2,8H_2O + H_2O\,(\text{solid}) \rightleftarrows H_2O \approx\approx x Cl_2.$$

(C.) Complete equilibrium between three solid phases. Examples unknown. (D.) Incomplete equilibrium between one solid phase and one liquid phase comprising two constituent substances. Examples:—A solution of Br_2H_2O (compare Roozeboom, *Rec. Trav. Chim.*, **5**, 362). (E.) Incomplete equilibrium between two liquid phases. Examples unknown.

(3.) Systems formed of more than two substances. (A.) Complete equilibrium between three solid and one liquid phase. Examples:—A double salt, its two constituents, and an aqueous solution. Examples

of complete equilibrium between (B) two solid and two liquid phases, (C) one solid and three liquid phases, and (D) four solid phases, are unknown. F. S. K.

Triple and Multiple Points regarded as Transition Points. By H. W. B. Roozeboom (*Rec. Trav. Chim.*, **6**, 304—332; compare preceding Abstract).—The transition point is the point of intersection of two curves of equilibrium for $n + 1$ phases of which n are equal, so that $n + 2$ phases can exist in this point. But these same $n + 2$ phases can be arranged in $n + 1$ systems of $n + 1$ phases, so that this same point must be a final point of $n + 1$ curves. For each of these equilibria, a single phase disappears when the system arrives at the point of transition.

The author discusses, with the aid of diagrams, the signification of the triple point in physical equilibria, and in cases of chemical equilibria, such as the conversion of rhombic into monosymmetric sulphur, classified according to the number of coexistent phases, and concludes as follows :—A triple point represents the only temperature and pressure which render possible the coexistence of three heterogeneous phases of the same substance. Three curves meet at this point representing the equilibria of the three systems of two phases which can be formed from these three phases. These curves divide the diagram (*p.t.*) into three regions, one for each phase. When heat is supplied to or taken from the system, we pass from the triple point on to one of the three curves by the disappearance of one of the phases owing to a transformation in which all three participate. In one direction, this phase is determinate, but in the other it may be one of the remaining two according to their proportionate quantities. The triple point is always a transition point in one direction for a single phase. Where one of the three phases at the triple point is a gas, this point may also be a transition point in the other direction, provided that the gaseous phase is in excess. The triple point is a limit of temperature for those phases which disappear entirely. There is one curve on one side of the triple point, and two on the other. The triple point is a transition point in the direction in which there is only one curve, and the phase which disappears is the one the region of which is comprised between the remaining two. The triple point is a limit for each of the three curves.

Quadruple Points.—Compare Roozeboom (*Rec. Trav. Chim.*, **5**, 393). If A, B, C are the three phases of two substances, and if the change which takes place at constant pressure when heat is supplied to or taken from the system, be expressed thus, $A \rightleftharpoons B + C$, from one point of the curve, it is only possible to pass to a higher temperature after the total disappearance of A. On the other hand, when heat is taken from the system, B + C are changed into A, but it will depend on their relative quantities which of the two phases will subsist with A to the left of the curve, A + B or A + C. A consideration of the transformation produced by change of pressure at constant temperature leads to the same conclusions, so that whereas there is only one region for a single phase on the two sides of a curve for two phases, the region of two of the three phases lies on one side

of a curve for three phases; the region for the two other systems of two phases lies on the other side. The author discusses, with the aid of diagrams, the signification of the quadruple point in equilibria classified according to the number of coexistent phases (compare Roozeboom, *Rec. Trav. Chim.*, **5**, 396), and concludes as follows:—The quadruple point is the only point (*p.t.*) in which four heterogeneous phases, into the composition of which two substances enter, can coexist in equilibrium. Four curves, each for three phases, meet in these points and divide the diagram (*p.t.*) into six regions for the six groups of two phases which can be formed. If heat is taken from or supplied to the system of four phases in the quadruple point, there is a transformation in which all four phases participate, and which terminates in the disappearance of one of them in each direction. The composition and specific volume of the phases must be known before the equation of this transformation can be given, as it takes place at constant volume. There are two cases possible: the equation may contain two phases on each side, or one phase on one side and three on the other. In the first case, there are two phases which can disappear in each direction, in the second case one phase only disappears in one direction and one of three phases in the other. When two or three phases can disappear, it depends on their proportionate quantities which does so. The quadruple point can be a transition point for, at the most, one phase in one direction, but it is not so always; and in the case of a liquid phase the quadruple point is a transition point only when the triple point is a transition point for a liquid between two limits of concentration. If one of the phases is gaseous, the quadruple point can always be a transition point for one of the other phases, provided that the gaseous phase is present in sufficient quantity. By the disappearance of one of the phases, when heat is taken from or supplied to the system, we pass from the quadruple point on to one of the four curves for three phases. Of these curves, there may be two on each side of the quadruple point, or one on one side and three on the other. In the first case, the quadruple point is an inferior limit of temperature for one system of two phases, and a superior limit for the other system of two phases. In the second case, the quadruple point is a limit for three regions of two phases situated on the same side and comprised between the three curves, whereas in the inverse direction it is a transition point for the phase which is common to these three regions. Each of the four curves of three phases which meets at the quadruple point has a limit.

Quintuple Points.—In a system of five phases formed of three constituent substances there is complete heterogeneous equilibrium when there are four coexistent phases. This equilibrium will be expressed by a curve (*p.t.*). The transformation which would take place at a point of such a curve, either by a variation of pressure at constant temperature or by a variation of temperature at constant pressure, would be completed by the disappearance of one of the four phases.

If A, B, C, D are the four phases, the transformation may take place thus: $A + B \rightleftharpoons C + D$; or thus: $A \rightleftharpoons B + C + D$. In the first case the phases $A + B + C$ and $A + B + D$ could exist on one side of

the curve, and the phases A + C + D and B + C + D on the other. In the second case A + B + C, A + B + D, and A + C + D on one side and B + C + D on the other. The curve can therefore divide the diagram into two regions for two systems of three phases, or into one region for one system and another in which three systems of three phases can exist. If two of these curves meet, there results a quintuple point in which three other curves terminate, together dividing the diagram into six regions for the ten groups of three phases which can be formed from the five phases coexisting in the quintuple point. The five curves can be situated to the right or to the left of the quintuple point 1 to 4 or 2 to 3, but the quintuple point is a transition point for a single phase only in the first case. F. S. K.

Osmotic Experiments with Living Membranes. By H. DE VRIES (*Zeit. physikal. Chem.*, **2**, 415—432).—This paper contains a description of the method used for determining the isotonic coefficients of solutions by observation of their behaviour towards plant-cells. The cells used are those of the leaves of *Tradescantia discolor*, *Curcuma rubricaulis*, and *Begonia manicata*. Viewed under the microscope, these cells are seen to consist of an outer sheath, which is rigid and defines the shape of the cell, being either tetragonal or hexagonal, containing the sap of the cell inclosed within an inner membrane. The outer sheath is permeable for water or aqueous solutions, the inner membrane for water alone and not for dissolved substances, fulfilling the conditions of a semi-permeable material. The inner membrane being elastic will follow by contraction or expansion any change in the volume of the sap which it incloses. In the normal condition, the sap, which is highly coloured for the above plants, fills the whole of the cell, the inner membrane being pressed closely against and indistinguishable from the inner side of the outer sheath. If the cell is immersed in an aqueous solution, this will permeate the outer sheath, and if its osmotic pressure is greater than that of the sap, water will pass through the walls of the inner membrane from the latter to the solution. The volume of the sap therefore decreases and a contraction will take place, the inner membrane leaving the sides of the outer sheath and being drawn together round the coloured sap, the spaces thus rendered vacant becoming filled with the colourless solution, a change easily observed under the microscope. By reducing the strength of the solution, the osmotic pressure decreases and the contraction may be brought down to a minimum; and finally when the osmotic pressure of the solution and the sap are the same, the contraction ceases to occur, and no change in the appearance of the cell takes place on immersion. A solution of this strength is then said to be isotonic with the sap, and of course those solutions isotonic with the sap of the same cells are isotonic with one another. The comparison of relations of the concentrations of isotonic solutions to one another gives their isotonic coefficients, that of potassium nitrate having been taken for physiological reasons as the standard and assumed equal to 3.

It must be pointed out that a cell immersed in a solution, the strength of which is below that of the isotonic, will undergo no

change in appearance, for, although water is absorbed by the sap, the rigidity of the cell walls prevents expansion taking place. In practice, it is customary to reduce the difference between the strengths of solutions which are observed to be above that of the isotonic and of those which are known to be beneath it to a minimum, and then to take the mean. The inner membranes were found to be practically impermeable for all the neutral substances examined, except that of the *Tradescantia* for glycerol, the exception here being due to some unknown physiological action. Acids, alkalis, and other substances which in any way chemically attack the cell cannot of course be examined.

A table is given of the isotonic coefficients of a number of substances; and a comparison of the same made with the molecular reduction of the freezing point and the vapour-tension, from which it appears that solutions of equal osmotic pressure have the same freezing point and the same vapour-tension. Osmotic pressure being, according to Van't Hoff, proportional to the molecular weight, a calculation of the extent to which dissociation of certain electrolytes in solution takes place is made, the results of which agree on the whole with those of Arrhenius. A determination of the molecular weight of raffinose gave for this the formula $C_{18}H_{32}O_{16},5H_2O$. H. C.

The Laws Governing the Reactions of Direct Addition. By I. KABLUKOFF (*J. Russ. Chem. Soc.*, 1887, 566—585).—When halogen hydrides unite with unsaturated hydrocarbons, the combination takes place according to a law which was first enunciated by Markownikoff as follows:—In the case of the combination of unsymmetrical hydrocarbons with halogen acids, the halogen combines with the carbon-atom in union with the smallest number of hydrogen-atoms, that is, with the carbon-atom which is most influenced by other carbon-atoms. The same rule holds good when the addition of sulphuric acid takes place. The author shows in several tables that the reactions depend on the heat of formation of the resulting compounds; the heat of formation of the tertiary alcohols and the corresponding compounds, for instance, is larger than that of their secondary and primary isomerides. When the halogen unites with the least hydrogenised carbon-atom, more heat is evolved than would be the case if it were added to the other side of the double linking. The reaction taking place is in accordance with Berthelot's principle of maximum of work.

According to Potilizin's investigations, however, the reaction which takes place at a higher temperature is the reverse of that which is found to take place at low temperatures. This is explained by a second empirical law of Markownikoff, namely: When an unsaturated molecule, C_mH_nX, combines with a saturated one, YZ, at a low temperature, the negative element or group will combine with the least hydrogenised carbon-atom, or with a carbon-atom already in union with a negative element or group, whereas at a higher temperature the reverse order of combination takes place. The addition of hypochlorous acid forms an exception, as it only in some cases follows this rule; sometimes the distribution of its elements takes place in both directions.

When organic oxides combine with the halogen hydrides, the halogen

combines with the least hydrogenised carbon-atom, and the hydroxyl-group is formed at the most hydrogenised carbon-atom. All these changes take place in accordance with the thermochemical data.

B. B.

Method for avoiding "Bumping" in Distillation. By W. MARKOWNIKOFF (*J. Russ. Chem. Soc.*, 1887, 520—521). The disagreeable phenomenon of "bumping" is avoided only for some little time after boiling commences when platinum wire or pieces of charcoal, &c., are introduced into the liquid. The author finds that good results are obtained on coating the inside of the vessel with a thin layer of silver; this, however, easily breaks off. A far better method is to introduce into the liquid a few very thin capillary glass tubes of 3 to 10 mm. in length, and sealed up at one end. The boiling then goes on quietly at ordinary or reduced pressure, even if a finely divided precipitate like barium sulphate occurs in an aqueous solution of a salt. Even such liquids as caustic soda may be boiled easily when these tubes are used.

Note.—The Abstractor has tried the device above suggested and found it invaluable, especially for the distillation of concentrated acids. He recommends that the capillary tubes be made of different sizes so that they may float in different layers of the liquid.

B. B.

New Apparatus. By C. LIEBERMANN (*Ber.*, **21**, 2528—2529).—1. *Apparatus for Fusion with Potash.*—The fusion is conducted in a nickel dish supported on a copper-bath constructed on the principle of V. Meyer's drying apparatus. The bath contains naphthalene, anthracene, or anthraquinone, &c., and is provided with a condenser.

2. *Desiccator for Substances Sensitive to Light.*—The desiccators are made of thick, brownish-yellow glass of the sort used for photographic dark rooms. Funnels, plates, and crystallising dishes of the same glass are also useful.

3. *Arrangement for Water-pumps.*—Nickel-plated water-pumps are screwed to one end of the bench; the outlet is soldered into a waste-pipe.

N. H. M.

Inorganic Chemistry.

Sulphur Compounds. By H. REBS (*Annalen*, **246**, 356—382).—Hydrogen persulphide, prepared by pouring aqueous solutions of potassium, sodium, or barium polysulphides into an excess of hydrochloric acid, always have the composition H_2S_5. The sp. gr. of the compound is 1·71 at 15°.

The author has obtained the compounds P_2S_5 and P_4S_3 in the crystalline form by heating a mixture of phosphorus and sulphur in the theoretical proportions and extracting the product with carbon bisulphide. The trisulphide, P_2S_3, is sparingly soluble in carbon bisulphide.

A comparison of the barium-derivatives of methyl and ethyl hydrogen sulphates prepared by (1) the action of water on methyl or ethyl sulphate, and (2) by the action of sulphuric acid on ethyl or methyl alcohol shows that the salts obtained from the different sources are identical. Church's statement (*Chem. Centr.*, 1856, 146) that the salts are isomerides is incorrect.

The author agrees with Weber (*Ann. Phys. Chem.*, **159**, 313) that Marignac's (*Ann. Phys. Chem.*, **139**, 480; *Jahresb.*, 1879, 209) isomeric modification of sulphuric anhydride is in reality a compound of the anhydride with a small quantity of water. W. C. W.

Dithionates. By K. Klüss (*Annalen*, **246**, 179—220 and 284—306).—When freshly precipitated bismuth hydroxide is treated with dithionic acid, an acid solution is formed which, on evaporation, deposits monoclinic crystals of the basic salt, $Bi_2S_2O_8 + 5H_2O$. If the acid solution is poured into water at 60°, a more basic salt, $4Bi_2O_3,3S_2O_5 + 5H_2O$, is precipitated.

Normal stannous dithionate can exist in solution, but has not been isolated. If the solution is poured into water, a basic salt, $8SnO,S_2O_5 + 9H_2O$, is deposited. The normal thorium dithionate, $Th(S_2O_6)_2 + 4H_2O$, is very unstable, and decomposes at the ordinary temperature with evolution of sulphur dioxide. Normal chromic dithionate, $Cr_2(S_2O_6)_3 + 18H_2O$, is obtained in octohedra when freshly precipitated chromic hydroxide is digested with a slight excess of dithionic acid. If the oxide is in excess, a green, amorphous basic salt, $3Cr_2O_3,4S_2O_5 + 24H_2O$, is produced. Uranium yields three basic dithionates of the composition $7UO_2,S_2O_5 + 8H_2O$; $6UO_2,S_2O_5 + 10H_2O$, and $8UO_2,S_2O_5 + 21H_2O$. Ammonium dithionate, $(NH_4)_2S_2O_6 + \frac{1}{2}H_2O$, crystallises in needles belonging to the monoclinic system. A basic beryllium salt, $5BeO,2S_2O_5 + 14H_2O$, is obtained as a gummy mass on carefully evaporating a solution of beryllia in dithionic acid.

Ferrous dithionate, $FeS_2O_6 + 7H_2O$, forms triclinic prisms. At 18·5° 1 part of salt dissolves in 0·59 of water. The normal ferric dithionate has not been isolated; a solution of the salt decomposes, yielding ferric sulphate, ferrous dithionate, sulphurous oxide, and sulphuric acid. If the solution is poured into water at 70°, a basic salt, $8Fe_2O_3,S_2O_5 + 14H_2O$, is deposited in the form of a brownish-red powder. Hydrogen peroxide converts ferrous dithionate into two basic salts, $3Fe_2O_3,S_2O_5 + 8H_2O$ and $Fe_2O_32S_2O_5$. The normal cobalt salt, $CoS_2O_6 + 8H_2O$, forms triclinic crystals. It is deposited from alcoholic solution in pink prisms containing only 6 mols. H_2O. Normal copper dithionate crystallises with 4 and also with 5 mols. H_2O. A basic salt, $4CuO,S_2O_5 + 4H_2O$, insoluble in water, is formed when the normal salt is digested with freshly precipitated cupric hydroxide. Another basic salt, $4CuO,S_2O_5 + 3H_2O$, is prepared by slowly raising the temperature of a dilute solution of copper acetate and sodium dithionate to 70°.

Mercuric dithionate, $HgS_2O_6 + 4H_2O$, is an unstable, crystalline compound. It is occasionally formed by digesting precipitated mercuric oxide in dithionic acid, but the exact conditions under which it is produced are not known. Aluminium dithionate, $Al_2(S_2O_6)_3 +$

$18H_2O$, forms transparent, deliquescent plates soluble in water and in alcohol. Dithionic acid does not act on ceric and thallic hydroxides at the ordinary temperature; at a higher temperature, the acid decomposes. A solution containing sodium and thallium in molecular proportion deposits rhombic crystals in which the proportion of Tl_2O to Na_2O is as 9 : 4. A mixture of lithium and thallium dithionates deposits monoclinic prisms, $Tl_2O : Li_2O = 3 : 4$.

The following mixed salts were also obtained: $2Tl_2S_2O_6,Ag_2S_2O_6$, apparently triclinic; $3Tl_2S_2O_6,2BaS_2O_6$; $2ZnS_2O_6,9(NH_4)_2S_2O_6 + 16\frac{1}{2}H_2O$, monoclinic; $ZnS_2O_6,5(NH_4)_2S_2O_6 + 9H_2O$; $CdS_2O_6,2(NH_4)_2S_2O_6 + 4\frac{1}{2}H_2O$, monoclinic; $FeS_2O_6,3(NH_4)_2S_2O_6 + 6H_2O$, monoclinic; and $2FeS_2O_6,9(NH_4)_2S_2O_6 + 16\frac{1}{2}H_2O$; manganese ammonium dithionate, $2MnS_2O_6,9(NH_4)_2S_2O_6 + 16\frac{1}{2}H_2O$, and the nickel and cobaltammonium salts are isomorphous with the zincammonium salt. The copper salt is not isomorphous, although it crystallises in the monoclinic system. Its formula is $2Cu_2S_2O_6,(NH_4)_2S_2O_6 + 8H_2O$. $Al_2(S_2O_6)_3 + (NH_4)_2S_2O_6 + 27H_2O$ forms deliquescent, monoclinic crystals. W. C. W.

Colloïds and the Water they Contain. By J. M. van Bemmelen (*Rec. Trav. Chim.*, **7**, 37—68).—A colloïd that feels dry still contains a considerable quantity of water, and when further dried at a constant temperature, the state of aggregation is continuously modified and with it the composition. When, at a certain temperature, a colloïd has lost so much water that its vapour-tension is equal to that of water at the same temperature, any further loss of moisture can be prevented by placing it in a closed space in proximity to water and keeping the temperature constant. If a colloïd, after exposure to dry air, has lost so much water that its vapour-tension has become practically null, it can regain its water either partially or entirely when placed in an atmosphere saturated with aqueous vapour, but the whole of the water can be regained only when the change in the state of aggregation is completely reversible. In a closed space partially saturated with moisture, a colloïd loses water until its vapour-tension is equal to that in the surrounding medium. The rapidity of dehydration in a dry medium at a constant temperature continually diminishes until it reaches a minimum, for instance, colloïdal metastannic acid containing 2·2 mols. H_2O, lost 0·55 mol. H_2O the first day, and this quantity gradually decreased to 0·01 mol. H_2O on the thirteenth day, when the composition was 0·79 mol. H_2O.

A colloïd loses or attracts moisture with decreasing rapidity until its vapour-tension is equal to that of water, and as a rule no definite hydrate is formed when it loses water. At every temperature, a colloïd attains a different final composition either in dry air or in an atmosphere saturated with moisture. The non-reversible modifications which most colloïds undergo in losing water, or when heated, complicate the process of dehydration or rehydration; generally the non-reversible modifications have a diminished absorptive power, but they retain their water with more energy. The vapour-tension is in this case more or less diminished.

When the temperature is raised, the decomposition of a colloïd is gradual; at first the vapour-tension increases and also the rate of

decomposition, it then decreases and reaches a minimum. The quantity of water in a colloïd either in a dry or a saturated atmosphere is less than what is present at a lower temperature, and the difference is greater the more the colloïd has undergone modification by the action of heat. A definite composition can be attained when a colloïd is heated sufficiently long at any temperature, but the more quickly, the higher the temperature, provided the initial composition is the same in both cases; for example, with colloïdal stannic acid containing about 3 mols. H_2O, the same composition (1 mol.) is attained in one hour at 103° or in several days at 15°. When the same quantity is heated successively at increasing temperatures, so long that at each temperature the decomposition attains a minimum, the rate of loss is found to vary. In the case of colloïdal alumina, the rapidity increases at about 220°, in the case of colloïdal glucina at 215°. These accelerations indicate a more intrinsic modification in the state of aggregation and are often accompanied by a change in colour.

Under favourable circumstances, colloïds can be transformed into definite hydrates. Crystalline glucinum hydrate (compare Bemmelen, *J. pr. Chem.* [2], **26**, 227) is only slightly hygroscopic; it has a definite composition which it retains even when heated at about 200°, at 204—215° slight decomposition occurs which becomes relatively rapid at 215—220°.

The compounds described by Ditte as hydrates of vanadic acid are colloïds.

For the transformation of a colloïd into a hydrate or chemical compound, it must undergo modification at a suitable temperature, such that it becomes insoluble in the medium in which it was dissolved when in the colloïdal state. F. S. K.

Colloïdal Silica. By J. M. VAN BEMMELEN (*Rec. Trav. Chim.*, **7**, 69—74).—Four samples of colloïdal silica prepared as follows:—*a*, by acidifying a solution of an alkaline silicate; *b*, by decomposing methyl orthosilicate with water; *c*, by decomposing silicon tetrachloride; and *d*, by decomposing silicon tetrafluoride with water, were quickly dried with blotting paper, and then heated until the weight was constant, at 15°, at 100°, and at a red heat (1), in ordinary air; (2), in dry air; and (3), in air saturated with moisture. The results obtained show that the four preparations retain different quantities of water at any given temperature, and that at every temperature equilibrium is established between the vapour-tension of the colloïd and that in the surrounding medium. *a* retains more water at 50° or even at 100° in a medium saturated with aqueous vapour that at 15° in dry air. *a* and *b* have the same vapour-tension as water at the ordinary temperature when they have absorbed about 4 mols. H_2O; *c* and *d* when they have absorbed 1·8 and 2·2 mols. respectively. This tension diminishes as the water is driven off, and approaches zero when the quantity of water present is $a = 0{\cdot}26$, $b = 0{\cdot}36$, $c = 0{\cdot}3$, and $d = 0{\cdot}3$ mols. *a* absorbs and loses water most readily, and is least modified by heat, inasmuch as its absorptive power is not diminished at all after it has been heated at 100°, and in a less degree than the other preparations after moderate calcining. *b* is more stable in the

dissolved state and retains its water with more energy than *a*, *c*, or *d* in dry air at 100°, but its absorptive power is greatly diminished after having been heated at 100°. *d* is most voluminous in the dry state, and retains the least quantity of water when exposed to the air at the ordinary temperature. *a*, *b*, and *c* entirely lose their absorptive power after having been calcined at a high temperature, but *d* still absorbs a small quantity of water at 15° in an atmosphere saturated with moisture.

The author concludes that the quantity of water retained by the colloïd between 15° and 100° in a damp atmosphere depends on the temperature, on the state of aggregation, and on the vapour-tension of the surrounding medium. F. S. K.

Colloïdal Alumina and Aluminium Hydrate. By J. M. VAN BEMMELEN (*Rec. Trav. Chim.*, **7**, 75—87; compare Ramsay, this Jour., 1877, ii, 396, and Carnelley and Walker, Trans., 1888, 54).—Four samples of colloïdal alumina were prepared as follows:—*a*″ by precipitating a solution of aluminium chloride with ammonia, washing for a long time and drying in the air; *a*′ by precipitating a very dilute solution of aluminium chloride (1 gram Al_2O_3 in 1 litre) with ammonia, washing as quickly as possible, and exposing to the air until dry to the hand; *b* by boiling *a*″ for 24 hours with a large quantity of water and then drying in the air; and *c* by keeping *a*″ for six months under water and then drying in the air.

After exposure to the air at the ordinary temperature, they have all practically the same composition, namely 4·3 to 4·5 mols. H_2O, but when placed at 15° in a dry atmosphere, or in an atmosphere saturated with moisture, or when heated at 100° in the air, in all cases until equilibrium is established, the quantity of water remaining is different in each sample in every case.

When *a*″ is heated at 100° in dry air, the quantity of water given off decreases from about 0·5 mol. to 0·02 mol. per hour, but the weight does not become constant even in nine hours. When the four samples are heated for 1 to 2 hours at temperatures increasing from 140° to 300°, the loss of water is different in each case, but it seems that this difference practically disappears between 250° and 300°, at which temperature they have all the approximate composition $Al_2O_3 + H_2O$. At any temperature between 15° and 300°, there is no increase or decrease in the rate of loss which would indicate the formation of a definite hydrate.

a′ retains and absorbs water with more force than *a*″ or *b*. Colloïdal alumina becomes more sparingly soluble in acids and alkalis in proportion to the quantity of water which it loses when heated; and after heating *a* and *a*″ at various temperatures, they absorb smaller quantities of water when placed in a saturated atmosphere, and they retain less in dry air in proportion to the loss of water which has occurred.

100 mols. of the dry colloïd, $Al_2O_3 + 5·5H_2O$, absorb 2·5 mols. of potassium sulphate when agitated at 50° with a solution containing 30 mols. of potassium sulphate in 100 c.c.; with the same weight of colloïd, the quantity of salt absorbed at a constant temperature in-

creases with the concentration and volume of the saline solution, but in a decreasing proportion.

The substance which separates from an alkaline solution of the colloïd, either spontaneously or by the slow action of carbonic anhydride, is a true hydrate and a definite chemical compound, $Al_2O_3 + 3H_2O$. It is crystalline under the microscope and is only slightly hygroscopic. It remains practically unchanged when heated at 160° in a stream of dry air; between 170° and 200° it is slowly decomposed but with decreasing rapidity, so that even after 30 hours it has not attained the composition $Al_2O_3 + 2H_2O$. Between 200° and 225°, the rate of decomposition increases and 1 mol. H_2O is driven off in a few hours, but the rate of loss gradually diminishes and the final composition attained is approximately $2Al_2O_3 + H_2O$. From 225° to 320°, decomposition is excessively slow, only at a temperature above 320° does the quantity of water fall to less than half a molecule in a relatively short time.

When it is heated for an hour in a bunsen flame, and then placed in a damp atmosphere, it absorbs 3·75 mols. H_2O in 13 days, but if then kept in dry air for six days, the water falls to about 2 mols.

This hydrate does not absorb crystalline substances from their solutions.

The sample *c* referred to above is in some respects similar to a true hydrate; after exposure to the air, its composition is approximately $Al_2O_3 + 3H_2O$. When heated at 100° to 170°, it loses water very slowly until the composition becomes about $Al_2O_3 + 2H_2O$, but it is amorphous and very hygroscopic. F. S. K.

Colloïdal Stannic Acid. By J. M. van Bemmelen (*Rec. Trav. Chim.*, **7**, 87—106).—The same quantity of colloïdal stannic acid was agitated for 15 minutes with the same volume of cold, dilute potash of various concentrations, and from the quantity of stannic acid dissolved and the subsequent behaviour of the solutions the author concludes that no chemical combination of stannic acid with potash is formed but only complex molecules, the composition of which varies considerably with the temperature and concentration.

Four samples of colloïdal stannic acid were prepared:—a^α by dialysing a hydrochloric acid or alkaline solution of stannic acid, or by the action of calcium carbonate on an aqueous solution of the chloride; a^β by passing a stream of carbonic anhydride through an alkaline solution; $a^\beta{}_2$ by the action of an atmosphere of carbonic anhydride, and $a^\beta{}_3$ by the action of ordinary air on an alkaline solution of stannic acid.

a^α has all the properties of a colloïd and appears amorphous even when magnified 750 times. A fifth sample, a^β, prepared by treating an alkaline solution of stannic acid with carbonic anhydride, also appeared amorphous under the same magnifying power; it is hygroscopic and has no definite composition.

The samples a^α, $a^\beta{}_1$, $a^\beta{}_2$ and $a^\beta{}_3$, contain different quantities of water when dry, and also after exposure to the air, or after keeping in a damp atmosphere. Colloïdal stannic acid has a vapour-tension equal to that of water at the ordinary temperature, when it contains about 3 mols. H_2O, but this quantity depends on the method of preparation,

as a^{β}_3 retains less than a^{β}_1. This tension approaches zero when the water falls to about 1 mol. When a^{α}, a^{β}_1, a^{β}_2 and a^{β}_3 are placed in dry air at about 15°, the vapour-tension and the rapidity of decomposition vary continually in all four samples, and if they are then placed in a damp atmosphere the water is only partially regained. a^{β}_2 retains more water at 55° in a damp atmosphere than at 15° in dry air; when heated at 55° in a moist atmosphere, it loses 1·12 mols. H_2O in $9\frac{1}{2}$ hours, and a further loss of 0·93 mol. takes place at 55° in dry air in 11 hours, but in both cases the rate of loss decreases with the time: a^{α} behaves similarly. At 100°, decomposition is more rapid in all the samples, but equilibrium is not established until after a long time, especially in the case of a^{β}_3.

If the colloïd has lost water by previous exposure to dry air or by heating at 55°, the decomposition at 100° takes place with greater rapidity. At 105°, 120°, and 160°, the decomposition is similar to that at 100°, the rate of loss increases with every rise of temperature and then decreases. The same ultimate composition can be attained at 15°, 55°, 100°, 105°, and 110°, but the lower the temperature the longer the time required.

Colloïdal stannic acid, prepared either by saturating a freshly prepared aqueous solution of stannic chloride with carbonic anhydride, or by saturating a freshly prepared potash solution of stannic chloride with carbonic anhydride, did not give the reactions of so-called metastannic acid, even after having been dried by pressure at the ordinary temperature, but after having been heated with water below 100°, or kept some time in a stoppered flask, so that no loss of water occurs, or when heated at 55° or 100°, it no longer dissolves entirely in concentrated hydrochloric acid, and gives in a greater or less degree the reactions of metastannic acid with nitric acid, sulphuric acid, and stannous chloride.

Colloïdal metastannic acid, dried at the ordinary temperature, contains 2·2 mols. H_2O, and absorbs very little water when placed in an atmosphere saturated with moisture; in dry air, the water falls to 0·8 mol., and the composition then remains practically constant. At 100°, the moisture falls to 0·68 mol. in one hour, and to 0·6 mol. after several hours.

The results obtained on heating colloïdal stannic acid and metastannic acid at temperatures increasing from 15° to 185°, show that the former is gradually changed into the latter when heated. When colloïdal metastannic acid, containing 2·3 mols. H_2O in an atmosphere saturated with moisture, has lost 1·5 mols. by exposure to dry air, it only regains 0·9 mol. when again placed in a saturated atmosphere; after having been heated at 100°, it only absorbs 0·7 mol.; and after having been calcined, it only regains 0·28 mol. When strongly calcined, it is no longer hygroscopic in ordinary air.

Colloïdal metastannic acid does not form any definite compound with potash when treated with solutions of various concentrations. The composition of the opalescent solutions obtained varies very considerably and is greatly influenced by the concentration; the undissolved colloïd absorbs some of the potash. Hydrochloric acid acts like potash, but the acid must be concentrated in order to form the

complex molecules which are again decomposed and dissolved on adding water, but are reformed and become insoluble when an excess of hydrochloric acid is added. Sulphuric acid precipitates the colloïd still more readily from its solution in water or dilute hydrochloric acid (compare *J. pr. Chem.* [2], **23**, 331—339). The yellow precipitate obtained with stannous chloride in a hydrochloric acid solution, and that produced by potassium ferrocyanide in a potash solution, are undoubtedly absorption compounds. F. S. K.

Colloïdal Ferric Oxide. By J. M. van Bemmelen (*Rec. Trav. Chim.*, **7**, 106—114).—The reddish-brown colloïd prepared by adding ammonia to a solution of ferric chloride, contains 6·2 mols. H_2O, after having been kept at 15° in an atmosphere saturated with moisture. After exposure to the air for a year, its composition is constant and it contains 4 to 4·1 mols. H_2O; in dry air, its composition becomes constant at 1·6 mol. after four months. When heated at 100° in dry air until equilibrium is established, it only contains 0·96 mol. H_2O, but the final composition depends on the state of aggregation. The same sample, after keeping for six years in a closed flask, was heated at temperatures varying from 15° to 150° in dry air and in a saturated atmosphere until the weight was constant; at 15° in dry air, it contained 1 mol. H_2O; and at 100°, 0·45 mol. H_2O after only five hours. Between 100° and 300°, the reddish-brown colloïd decomposes slowly and regularly until the water falls to about 0·25 mol., but if heated sufficiently long at any intermediate temperature the final result is practically the same. When placed in an atmosphere saturated with moisture, after having been heated at temperatures varying from 100° to 300°, it only partially regains the water it has lost; and the higher the temperature to which it has been exposed the greater is the diminution in absorptive power: after having been calcined it absorbs no moisture. This reddish-brown substance is therefore a colloïd of variable composition. The yellow colloïd prepared by Phillips's method contains 2·43 mols. H_2O when dry, but when kept for a month in a saturated atmosphere it contains 7·4 mols. H_2O. When heated at temperatures increasing from 15° to 270° in a stream of dry air, the water falls from 1·7 to 0·24 mol. Above 200°, its composition is practically the same as that of the reddish-brown colloïd at the same temperature; but from about 50° to 300° the former retains water with more energy. F. S. K.

Colloïdal Chromic Oxide. By J. M. van Bemmelen (*Rec. Trav. Chim.*, **7**, 114—118).—The bluish colloïd, prepared by treating a dilute solution of a chromic salt with ammonia at the ordinary temperature or at 100°, contains originally 11 mols. H_2O; after exposure to the air at 15° it contains 7·8 to 8 mols., but after keeping for 14 days in an atmosphere saturated with moisture, the quantity of water rises to 13·2 mols., and after exposure to dry air it falls to 7·0 mols. When heated in the air at temperatures increasing from 45° to 200°, the water falls from 5·9 to 2·3 mols., and the colour changes to a dirty green. The results obtained on heating at temperatures varying from 15° to 100° (1) in a saturated atmosphere, (2) in ordinary air,

and (3) in dry air, until the weight is constant, show that at every temperature equilibrium is established between the vapour-tension of the colloïd and that of water at the same temperature. At 65° and at 100°, it retains more water in a saturated atmosphere than at 15° and 65° respectively in a dry atmosphere. After having been heated at from 15° to 100° in dry air, its absorptive power is only slightly diminished, and at higher temperatures it retains more water than colloïdal silica, alumina, stannic acid or ferric oxide at the same temperature, but the higher the temperature to which it has been exposed, the more insoluble it becomes in acids and especially in alkalis. Colloïdal chromic oxide has no definite composition at any temperature between 15 and 280°. F. S. K.

Ultramarine Blue. By F. Knapp (*J. pr. Chem.* [2], **38**, 48—64; compare Abstract, 1887, 110).—When an intimate mixture of equal parts of pure sodium carbonate and flowers of sulphur is heated in a glass tube with a bunsen burner until no more sulphur sublimes, the weight of the residue, from 100 grams of sodium carbonate, is 167·9 grams on the average. If the temperature is raised, the weight remains constant for some time, but at a red heat sulphur again sublimes, and the weight of the residue is 150·6 grams on the average. No further evolution of sulphur occurs even when the mixture is heated with the aid of a blowpipe.

An aqueous solution of liver of sulphur deposits a black precipitate which decomposes in dilute solutions; if this precipitate is washed by decantation as long as the solution is sufficiently concentrated to prevent decomposition, and then boiled with potassium cyanide to free it from ferrous sulphide, it becomes stable, and can be washed with water. This substance is the black modification of sulphur (Magnus), and is the colouring principle of liver of sulphur. When sulphur is gradually added to fused sodium chloride, potassium chloride, or sodium sulphate, and the heating continued until the black turbidity which forms is just dissolved, a bright blue mass is obtained.

The production of ultramarine blue by the method described (*loc. cit.*) is explained as follows:—When liver of sulphur is heated, sulphur is liberated, and, coming in contact with surfaces at a temperature far above its melting point, is converted into the black modification which dissolves in the sodium sulphide and gives to it its greenish tinge. When the sulphide is dissolved in water and the solution brought into contact with calcium phosphate, the black modification is precipitated and the blue produced. Alumina, silica, kaolin, &c., have not sufficient surface attraction until they have become coated with sodium sulphide, so that the blue colour is not formed until the mixture is heated.

Potassium sulphide cannot be employed for the preparation of ultramarine blue, because the black modification of sulphur which is formed does not dissolve but separates at the surface and gets oxidised. F. S. K.

New Method of Forming Alloys. By W. Hallock (*Zeit. physikal. Chem.*, **2**, 378—379).—The author finds that alloys may be

formed from their components at a temperature below that at which the metal of lowest melting point melts, and without extra pressure, provided the temperature be above the melting point of the alloy itself. An instance of this is Wood's alloy, which is formed from 1 part of cadmium, 1 part of tin, 2 parts of lead, and 4 parts of bismuth; by mixing the metals in the finely-divided state, bringing the mixture into a tube with just sufficient pressure to insure the particles being together, and then heating for some time at 98—100°. Tin and lead may be alloyed by simply laying a clean surface of tin on a clean surface of lead and heating for some hours at 198—200°. The alloy of potassium and sodium which melts above 6° is made by cutting clean surfaces of the metals and pressing them lightly together at the ordinary temperature. H. C.

Astracanite and Hydrated Double Salts. By H. W. B. Roozeboom (*Rec. Trav. Chim.*, **6**, 333—355).—The transition points which Van't Hoff has described for the formation or decomposition of astracanite, sodium ammonium racemate, and cupri-calcic acetate, are quintuple points (compare this vol., p. 1152). In the case of astracanite the following phases can coexist at 22°:—$Na_2SO_4,10H_2O$(N), $MgSO_4,7H_2O$(M) astracanite = $Na_2Mg(SO_4)_2,4H_2O$(A), a solution having the composition $100H_2O \approx \begin{matrix} 4{\cdot}6MgSO_4 \\ 2{\cdot}9Na_2SO_4 \end{matrix}$(L) (see below), and aqueous vapour (G). At the quintuple point, there are, therefore, five phases composed of three substances, H_2O, $MgSO_4$, and Na_2SO_4. When heat is supplied to the system, M + N are transformed into A + L. This phenomenon being accompanied by expansion, and the pressure remaining constant as long as the five phases co-exist, part of the aqueous vapour must also dissolve, so that the transformation is expressed thus: N + M + G $\rightleftarrows$ A + L; but this equation only gives the symbols of the five phases; in order to express their quantities, the density of the aqueous vapour and of the solution at the quintuple point must be known. The equation shows that the transformation produced by heat will terminate in the disappearance of one of the phases, N, M, G, according to their quantities. From this moment we pass from the quintuple point to higher temperatures with one of the following systems of four phases: A + L + N + M, A + L + N + G, A + L + M + G. This necessitates the existence of three curves for these three complete heterogeneous systems, to the right of the quintuple point. On the other hand, when heat is taken from the system of five phases at the quintuple point, the transformation of A + L into N + M + G may terminate either in the disappearance of A or of L, and there would be two curves for the systems N + M + A + G and N + M + L + G, to the left of the quintuple point. This point is not, therefore, a transition point for only one phase. It is a transition point in the direction ← for the system of two phases, A + L, and these two phases could never exist at any temperature lower than the quintuple point, whereas every other group of two phases amongst M, N, A, L, G could exist on both sides of this point. The quintuple point is also a maximum temperature for the group of phases M + N + G, which could not

exist above this temperature, whereas every other group of three phases amongst N, M, A, L, G could exist on both sides of the quintuple point.

The author discusses, with the aid of diagrams, the case of astracanite and cupri-calcic acetate (compare *Rec. Trav. Chim.*, **6**, 91; Abstr., 1888, 401), and arrives at the following conclusions:—

The quintuple point of hydrated double salts is only a limit of temperature for those salts which contain a greater number of molecules of water than their constituent parts, and of which the solution and decomposition into these constituents is accompanied by contraction.

If these two conditions are not both satisfied, the double salt can exist on both sides of the quintuple point in presence of aqueous vapour or of a solution.

A large quantity of astracanite, or of a mixture of the double salt with one of its constituents, was agitated for about three hours in a closed vessel at constant temperature with a quantity of water so small that on standing only a very thin stratum of solution was obtained.

A certain volume of the solution was analysed, and the results are given in the following table:—

Temperature.	Astracanite.		Astracanite + sodium sulphate.		Astracanite + magnesium sulphate.	
	Na_2SO_4.	$MgSO_4$.	Na_2SO_4.	$MgSO_4$.	Na_2SO_4.	$MgSO_4$.
18·5°	—	—	—	—	3·41	4·27
22·0	2·95	4·70	2·95	4·70	2·85	4·63
24·5	3·44	3·70	3·45	3·62	2·75	4·71
	3·46	3·66			2·62	4·81
30·0	3·60	3·60	4·58	2·91	2·30	5·31
	3·59	3·59				
35·0	3·69	3·69	4·30	2·76	1·75	5·89
	3·74	3·74			1·71	5·87
47·0	3·60	3·60	—	—	—	—

From the diagram which is given, showing the curves of solubility at various temperatures, it is seen that above 25° a solution prepared from astracanite and an excess of sodium sulphate contains a greater number of molecules of sodium sulphate than of magnesium sulphate, but below 25°, even in the presence of $Na_2SO_4,10H_2O$, a solution is obtained containing a larger number of molecules of magnesium sulphate than of sodium sulphate. It is evident, therefore, that if the pure double salt is placed in contact with water it will be partially decomposed, and $Na_2SO_4,10H_2O$ will separate until equilibrium is established. The lower the temperature, the greater will be the decomposition, until at 22° the same solution is obtained from pure astracanite and from a mixture of the double salt and sodium sulphate.

A saturated solution of astracanite and magnesium sulphate always

contains a larger number of molecules of magnesium sulphate than of sodium sulphate, so that such a solution can never be obtained from astracanite alone. The values obtained at 18·5° with a solution prepared from astracanite and magnesium sulphate show that, if the double salt could be kept from decomposing at still lower temperatures, at about 15°, a solution would be obtained containing an equal number of molecules of the two sulphates. At 22°, the double salt could exist in presence of $Na_2SO_4,10H_2O$ and $MgSO_4,7H_2O$ at the bottom of a solution containing 4·65 $MgSO_4$ and 2·9 Na_2SO_4 in 100 H_2O. It is this solution which exists at the quintuple point (see above). At 17·9°, the solution contains 2·12 Na_2SO_4 and 4·56 $MgSO_4$, at 18·5° 2·16 Na_2SO_4 and 4·59 $MgSO_4$ (compare Van't Hoff, *Rec. Trav. Chim.*, **5**, 259, and Diacon, *Jahrsb.*, 1866, 64). At the transition point (22°) for the double salt in presence of a solution, the solubility is the same for each of the three systems: astracanite + $MgSO_4$, astrakanite + Na_2SO_4, and Na_2SO_4 + $MgSO_4$.

At temperatures above 25°, astracanite, placed in contact with water, dissolves without either of the simple salts separating, because the solution which is obtained in presence of either constituent contains a larger quantity of this constituent than is present in the double salt. From 25° to 47°, the solubility of astracanite hardly varies at all with the temperature.

The relation between the molecular quantities of sodium sulphate and magnesium sulphate in the different solutions which can exist in equilibrium with the double salt between 22° and 35° (?) can probably be expressed very closely by the formula $C_{MgSO_4} \times C_{Na_2SO_4} = K$. This product varies very slightly with the temperature.

Two facts of general importance result from this investigation, namely—(1) There are two limits of composition for the solution which can exist in equilibrium with a double salt. These limits are reached when the liquid is placed in contact with the double salt and with each of its constituents. (2) There is a limit of temperature above or below which the double salt is either decomposed or not by the solvent. F. S. K.

Chlorosulphides of Mercury. By T. Poleck and C. Goercki (*Ber.*, **21**, 2412—2417).—When chlorine is passed into water containing freshly precipitated mercuric sulphide, the latter becomes bright yellow, and finally dissolves. The bright yellow substance has the composition $2HgS,HgCl_2$ (compare Rose, *Ann. Phys. Chem.*, **13**, 59). When solutions of sodium thiocarbonate and mercuric sulphide are mixed, the following reaction takes place:—

$$HgCl_2 + Na_2CS_3 + 2H_2O = HgS + 2NaCl + CO_2 + 2H_2S,$$

but if a large excess of dry mercuric chloride is treated with a solution of sodium thiocarbonate, the product has the composition $2HgS,HgCl_2$. This same substance is formed when a solution of mercuric chloride (2 mols.) is precipitated with a solution of sodium thiosulphate (1 mol.).

When mercuric sulphide (1 mol.) is treated with a solution of mercuric chloride (1 mol.), half of the latter remains in solution,

whereas when 2, 3, 4, or 5 mols. of the sulphide are present the solution contains no chloride, and bright yellow products are formed, the compositions of which are $2HgS,HgCl_2$, $3HgS,HgCl_2$, and $4HgS,HgCl_2$ respectively. These products are all insoluble in hydrochloric acid, nitric acid, and water, but are blackened and decomposed by potash. They can also be obtained by boiling a solution of mercuric chloride (1 mol.) with 2, 3, and 4, mols. of the sulphide. They are not by boiling with a concentrated solution of changed sodium chloride, but at 190° they are converted into their constituents. When heated at 170° with a solution of potassium iodide, the substance $2HgS,HgCl_2$ is decomposed according to the equation—

$$2HgS,HgCl_2 + 4KI = 2HgS + HgI_2,2KI + 2KCl,$$

and the other products behave similarly. In all cases, the red modification of mercuric sulphide is obtained, which fact the authors consider is a proof that these products are true compounds.

Freshly precipitated mercuric sulphide is not converted into the red modification when treated in the same way, nor is the product obtained by precipitating mercuric chloride with sodium thiosulphate.

F. S. K.

Organic Chemistry.

Formation and Decomposition of Ethereal Salts: Compounds of Amylene (Trimethylethylene) with Acids as Cases of Chemical Equilibrium. By D. Konowaloff (*Zeit. physikal. Chem.*, **2**, 380—389).—The compounds of amylene with acetic acid and its chloro-derivatives, as has already been shown (this vol., p. 340), undergo decomposition when heated in the presence of any of the free acid, although otherwise stable. The decomposition is limited by the amount of free acid present, the temperature, and the time of heating. On the other hand, from experiments with the dichloracetate, it would appear that the presence of excess of the acid is more favourable to the formation of the salt than the presence of excess of the amylene, which appears rather to retard the reaction. This retardation caused by the excess of amylene present, is, as the author thinks, due to a change in the condition of the acid which the amylene affects. Such a change would be similar to that observed by Raoult in acetic acid, the molecular weight of which, when determined by the lowering of the freezing point, is double in benzene what it is in formic acid. This is supported by the fact that if trichloracetic acid be treated with excess of amylene, the formation of the salt takes place more readily on the addition of benzene to the solution, and that with a large quantity of benzene present the conditions of the reaction are very nearly those required by the law of mass action.

H. C.

The Hydrocarbons C_8H_{16} and C_9H_{18} obtained from Methyl Dipropyl Carbinol and Ethyl Dipropyl Carbinol. By E. SOKOLOFF (*J. Russ. Chem. Soc.*, 1887, 599—601).—The hydrocarbon C_8H_{16} was obtained when methyl dipropyl carbinol was prepared from methyl iodide, zinc, and butyrone. It is a light, mobile liquid, boiling at 120·4° (corr.), and having the sp. gr. 0·73138 at 20°; 0·72287 at 30°; and 0·71746 at 35°. The bromide is $C_8H_{16}Br_2$. The products of oxidation are propionic and acetic acids. The probable constitution is CMePr:CHEt. The hydrocarbon, C_9H_{18}, was obtained from the iodide of ethyl propyl carbinyl and alcoholic potash. It boils at 138°, and has the sp. gr. 0·74333 at 20°; 0·74344 at 30°; and 0·72990 at 35°.

B. B.

Isomeric Change of Disubstituted Acetylenes and of Dimethylallene under the Influence of Metallic Sodium: Synthesis of Acetylenecarboxylic Acids. By A. FAVORSKY (*J. Russ. Chem. Soc.*, 1887, 553—565).—Disubstituted acetylenes are usually said to be distinguished from their monosubstituted isomerides containing the acetylene group CH:C, by not yielding copper and silver compounds, as the latter do, and by not uniting with the alkali metals. The author finds that this is not strictly accurate, as the disubstituted acetylenes unite easily with sodium, forming metallic derivatives, which are usually obtained in the form of white powders. When treated with alcohol or water, these compounds yield oily substances, which have the odour and other properties of monosubstituted acetylenes, forming characteristic copper and silver compounds, and yielding the corresponding ketones on treatment with sulphuric acid. The author has examined the question whether the intramolecular or isomeric transformation takes place when the sodium acts on the hydrocarbon or when the sodium compound is decomposed by alcohol or water. In the first place, the sodium-derivatives were investigated in the case of methylethylacetylene, methylpropylacetylene, and dimethylallene.

Methylethylacetylene was sealed up with slightly less than its equivalent of sodium in a tube, and heated at 100°. The complete formation of the sodium compound takes place easily, and no pressure was observed on opening the tube. The product was washed with ether, and decomposed by water, when propylacetylene was formed; this gives precipitates with ammoniacal solutions of copper and silver nitrates. On heating it in sealed tubes with mercuric chloride, methyl propyl ketone is obtained. In order to ascertain the constitution of the sodium compound it was mixed with ether and dry carbonic anhydride was passed over it for a day, when it was found to be converted into the sodium salt of a carboxylic acid. The acid itself, set free by means of dilute sulphuric acid, forms feathery crystals melting at 27°, and distilling only under reduced pressure (at 125° and 20 mm.). It is deliquescent, although not easily soluble in water, but easily soluble in alcohol, ether, and light petroleum. The vapour-density of the acid could not be determined, as on boiling it is decomposed into propylacetylene and carbonic anhydride. Its composition is $CH_2Me{\cdot}CH_2{\cdot}C{:}C{\cdot}COOH$, and the following salts are described:— $C_6H_7NaO_2$; $(C_6H_7O_2)_2Ba + 3H_2O$; $(C_6H_7O_2)_2Ca$; $(C_6H_7O_2)_2Cu + 2H_2O$.

The silver salt could not be obtained. On acting with silver nitrate on the ammonium salt, a decomposition of the precipitate first formed takes place, carbonic anhydride being produced, together with the silver compound of valerylene, which yields propylacetylene when treated with weak hydrochloric acid. The acid is therefore *propylacetylenecarboxylic acid*, and the original sodium compound has the constitution $CH_2Me{\cdot}CH_2{\cdot}C{:}CNa$. The formation of this latter without evolution of hydrogen may be explained by the following equation:—

$$3CH_2Me{\cdot}C{:}CMe + 2Na = 2CH_2Me{\cdot}CH_2{\cdot}C{:}CNa + CH_2Me{\cdot}CH{:}CHMe.$$

Dimethylallene under similar circumstances yields a sodium-derivative, which on decomposition with water gives isopropylacetylene. The sodium compound is converted by carbonic anhydride into a sodium salt, from which *isopropylacetylenecarboxylic acid*,

$$CHMe_2{\cdot}C{:}C{\cdot}COOH,$$

was obtained. The calcium, barium, and copper salts are described. Instead of the silver salt, valerylene silver was formed, as in the case of the propyl-derivative; this gave isopropylacetylene on treatment with weak hydrochloric acid. The acid melts at 38°, and boils at 106—107° under 20 mm. pressure. The constitution and formation of the sodium compound may be represented as follows:—

$$3CMe_2{:}C{:}CH_2 + 2Na_2 = 2CHMe_2{\cdot}C{:}CNa + C_5H_{10}.$$

The sodium compound of *methylpropylacetylene* was prepared by heating the hydrocarbons with sodium at 150—160°. When treated with water, it gives butylacetylene and carbonic anhydride. *Butylacetylenecarboxylic acid*, $CH_2Me{\cdot}CH_2{\cdot}CH_2{\cdot}C{:}C{\cdot}COOH$, is a liquid which boils at 135° under 20 mm. pressure, and does not solidify at −20°. The barium and calcium salts are described. The silver salt is a little more stable than the salts of the acids previously mentioned, but after a time it decomposes in a similar manner. The change is analogous to those described above: $3CH_2Me{\cdot}CH_2{\cdot}C{:}CMe + 2Na = CH_2Me{\cdot}CH_2{\cdot}CH_2{\cdot}C{:}CNa + C_6H_{12}$. Under the influence of alcoholic alkalis, monosubstituted acetylenes are, as the author has found (this vol., p. 798), converted into the disubstituted ones, whereas the reverse reaction is now shown to take place under the influence of sodium. In this process the formation of any intermediate compounds cannot be assumed, and the reaction may therefore be regarded as a typical case of a change of the position of atoms within the molecule.

B. B.

Methyl Chlorothioformate: Polymeric Thiocarbonyl Chloride. By B. Rathke (*Ber.*, **21**, 2539—2545).—The polymeride obtained by exposing thiocarbonyl chloride to light melts at 116° (not 112·5°). When the solution in benzene is heated in a water-bath with aniline, the compound $S<\begin{matrix}CCl_2\\CS\end{matrix}>NPh$ is formed. This crystallises in lustrous, rhombic plates, melts at 69·5°, and is readily soluble in the usual organic solvents. When the polymeride is heated with 3 mols. of aniline dissolved in benzene, for half an hour, and then again with the same amount of aniline, phenylthiocarbimide, diphenylthiocarbamide, and the hydrochlorides of aniline and tri-

phenylguanidine are formed. These reactions make it probable that the polymeride has the constitution $CSCl{\cdot}S{\cdot}CCl_3$. This is confirmed by the similar properties of the corresponding oxygen-derivative, methyl perchloroformate (Hentschel, Abstr., 1887, 1027 and 1099).

The compound $C_2S_2Cl_4$ is converted by aqueous ammonia into ammonium thiocyanate and chloride. It is decomposed by cold alcohol, with formation of an oil. N. H. M.

Action of Methyl Iodide and Zinc on Ethyl Propyl Ketone. By E. Sokoloff (*J. Russ. Chem. Soc.*, 1887, 587—595).—For the investigation of Saytzeff's reaction in the case of mixed ketones, ethyl propyl ketone was mixed with methyl iodide and finely granulated zinc added. After standing for 11 days in water at the ordinary temperature, the mixture was heated for three days on a water-bath. *Methyl ethyl propyl carbinol*, $CMeEtPr{\cdot}OH$, was obtained as an aromatic liquid boiling at 140·3°, and having a sp. gr. of 0·8234 at 20° and 0·8106 at 35°. On heating it with acetic anhydride at 140°, the acetate, $CMeEtPr{\cdot}OAc$, boiling at 158—159°, was obtained.

Chromic acid oxidises the alcohol to propionic and acetic acids, carbonic anhydride, some ethyl propyl ketone being formed at the same time, together with a small quantity of a hydrocarbon, C_7H_{14}, boiling at 97·4°, and having the sp. gr. 0·71812 at 20°, 0·70879 at 30°, and 0·70535 at 35°. It yields the bromide, $C_7H_{14}Br_2$, and the iodide, $C_7H_{14}I_2$. The coefficient of refraction found corresponds with one double union in the hydrocarbon. On oxidising this hydrocarbon with chromic mixture, propionic and acetic acids were obtained without any ketone; its constitution, therefore, remains undetermined.

B. B.

Action of Ethyl Iodide and Zinc on Ethyl Propyl Ketone. By E. Sokoloff (*J. Russ. Chem. Soc.*, 1887, 595—598).—The method employed was the same as that described in the former paper (preceding Abstract). *Diethyl propyl carbinol*, $CEtPr{\cdot}OH$, was obtained, boiling at 160·5°, and having the sp. gr. 0·83794 at 20°, 0·83008 at 30°, and 0·82615° at 35°. The acetate boils at 176—178°. The carbinol, when oxidised, yields butyric, propionic, and acetic acids. An unsaturated hydrocarbon, C_8H_{16}, is formed at the same time; this boils at 118·5°, and has the sp. gr. 0·73465 at 20°, 0·72598 at 30°, and 0·72034 at 35°. The bromide is $C_8H_{16}Br_2$. The oxidation products of the hydrocarbon are butyric, propionic, and acetic acids. Constitution unknown. B. B.

Butallyl Methyl Pinacone. By I. Kablukoff (*J. Russ. Chem. Soc.*, 1887, 513—516).—This is obtained as a bye-product in the preparation of butallyl methyl carbinol by Crow's method from allyl acetone, addition of hydrogen taking place. Its formula is $C_{12}H_{22}O_2$, or $C_3H_5{\cdot}CH_2{\cdot}CMe(OH){\cdot}CMe(OH){\cdot}CH_2{\cdot}C_3H_5$. It is an oily liquid boiling at 180—182° (74 mm. pressure), or with partial decomposition at 264·5—266·5° (760 mm.). Its sp. gr. is 0·9632 at 0°, and 0·9452 at 24°. The coefficient of dilatation between 0° and 24° is 0·0008238. By the action of bromine, the compound $C_{12}H_{22}Br_4O_2$, is formed: an additive compound corresponding with the saturation of

the two allyl-groups. On oxidation with potassium permanganate in alkaline solution, ethyl acetate is regenerated, the analogy with pinacone being thus shown. B. B.

Derivatives of Hexyl Glycerol (Hydrated Butallyl Methyl Carbinol Oxide). By I. KABLUKOFF (*J. Russ. Chem. Soc.*, 1887, 502—513).—This glycerol was described by Markownikoff and the author in 1880 as the first homologue of glycerol in the fatty series. Before giving an account of some of its derivatives, the author proposes a new nomenclature for glycerols, the possible number of isomerides of glycerol with six carbon-atoms, for instance, being more than 20. The name of a glycerol is derived from the name of the unsaturated alcohol from which it is obtained, whilst the glycides are regarded as the oxides of these alcohols, and the glycerols as their hydrates.

The glycerol in question corresponds with butallyl methyl carbinol, $CH_2{:}CH{\cdot}CH_2{\cdot}CH_2{\cdot}CHMe{\cdot}OH$; its oxide is

$$<\!\overset{CH_2}{\underset{O}{\text{—}}}\!>CH{\cdot}CH_2{\cdot}CH_2{\cdot}CHMe{\cdot}OH,$$

and the hydrate of this oxide, $CH_2(OH){\cdot}CH(OH){\cdot}CH_2{\cdot}CH_2{\cdot}CHMe{\cdot}OH$, is hexyl glycerol.

Hexyl Glyceryl Ketone.—In order to prepare this hexyl ketone, sodium is dissolved in alcohol and ethyl acetoacetate and epichlorhydrin added. After heating the mixture on the water-bath, the sodium chloride is removed by filtration, the alcohol distilled off under diminished pressure, and water is added, when an oily layer separates at the bottom of the aqueous liquid; by agitating the aqueous liquid with ether, a further quantity of the oil may be extracted. This oil was saponified with baryta, and the excess of the latter separated by passing in carbonic anhydride. After evaporation on the water-bath, a thick, oily liquid was obtained, from which some baryta salt separated on the addition of absolute alcohol, and the alcoholic extract yielded the ketone of hexyl glycerol, $C_6H_{10}O(OH)_2$. This boils at 170—180° under a pressure of 15 mm., but was not further investigated.

The Monochlorhydrin of Hexyl Glycerol.—This may be prepared by Eltekoff's method, namely, the action of aqueous hypochlorous acid on butallyl methyl carbinol, but as the yield is very small, only a small part of the carbinol entering into reaction, Lauch's method (Abstr., 1885, 1194) is to be preferred. For this purpose, the carbinol (10 grams) is mixed with a saturated solution of boric acid (200 c.c.) and a solution of bleaching powder gradually added. After standing in the dark for 24 hours, the product is extracted with ether, when the chlorhydrin, $C_6H_{13}ClO_2$, is obtained (5·5 grams). It can be distilled under reduced pressure, but under atmospheric pressure (at 180°) it is decomposed into the glycide and hydrochloric acid. The *monochlorhydrinacetin* is obtained on addition of hypochlorous acid to butallyl methyl carbinol acetate and extraction with ether. It can be distilled only under reduced pressure, when it boils at 172—176° (50 mm.).

University of Michigan

The Glycide of Hexyl Alcohol (Butallyl Methyl Carbinyl Oxide).—This is formed by the action of potassium hydroxide (2 mols.) on the monochlorhydrinacetin at a gentle heat. It is a light, mobile liquid of sp. gr. 1·0099 at 21°, boiling at 178—181°. It has the formula $<^{CH_2}_{O—}>CH\cdot CH_2\cdot CH_2\cdot CHMe\cdot OH$, but it cannot be got quite free from chlorine compounds. It is not altered by heating with water at 115° for 44 hours. The author did not succeed in adding the elements of hydrogen chloride to the glycide so as to convert it into the monochlorhydrin. In order to obtain the ketone of the glycerol, $C_6H_{11}(OH)_2O$, allylacetone is treated with hypochlorous acid. The product is saponified with baryta, the excess of the latter removed by carbonic anhydride, and after removing the barium carbonate by filtration, the liquid is evaporated on the water-bath. The residue is then extracted with alcohol, and the alcoholic extract evaporated, when a liquid is obtained which may be distilled under reduced pressure. The oil thus obtained has the formula

$$<^{CH_2}_{O—}>CH\cdot CH_2\cdot CH_2\cdot CO\cdot CH_3,$$

that is, it is the oxide of allylacetone, the expected ketone.

B. B.

Action of Polyatomic Alcohols on Solutions of Boric Acid and Hydrogen Sodium Carbonate. By C. Jehn (*Arch. Pharm.* [3], **26**, 495—497; compare Abstract, 1887, 790).—Sulzer has found that solutions of boric acid or borax and normal sodium carbonate react on each other on the addition of glycerol and honey (glucose), provided the boric acid or borax solution be added in considerable excess. The author confirms the above, and finds further that chemically pure lævulose causes reactions in solutions of $NaHCO_3$ and H_3BO_3, in $NaHCO_3$ and $Na_2B_4O_7$, and in Na_2CO_3 and H_3BO_3 (in this case under Sulzer's conditions, when mannitol also is effective). Solutions of borax and normal sodium carbonate do not react either on addition of lævulose or mannitol. The alcohol dulcitol (melampyrit), $C_6H_8(OH)_6$, and the aldehydes lævulose and lactose cause the reaction. The alcohol quercitol, $C_6H_7(OH)_5$, does not influence the mixed solutions. The author concludes that the reaction is caused by those polyatomic alcohols and corresponding aldehydes only where their molecules contain as many hydroxyl-groups as carbon-atoms.

J. T.

Propylenediamine and Trimethylenediamine. By H. Strache (*Ber.*, **21**, 2358—2369).—Propylenediamine hydrochloride melts at 220° (compare Hofmann, *Ber.*, **6**, 308).

A compound, $C_6H_{14}N_2 + H_2O$, is contained in the higher boiling portions of the product obtained by the action of propylene bromide on alcoholic ammonia. It is a slightly yellow, mobile oil, boils at 203—207°, and is miscible with water, alcohol, and benzene in all proportions. It is not decomposed by alkalis or chromic acid, and it combines readily with methyl iodide.

Diacetylpropylenediamine, $C_3H_6(NHAc)_2$, prepared by adding an

excess of acetic anhydride to aqueous propylenediamine, crystallises from alcoholic benzene in white, deliquescent needles, melts at 138—139°, and is readily soluble in water, alcohol, and chloroform, sparingly in benzene, and insoluble in ether and light petroleum.

Dibenzoylpropylenediamine, $C_3H_6(NHBz)_2$, prepared by heating a mixture of benzoic chloride and aqueous propylenediamine, separates from benzene in colourless, granular crystals, melts at 192—193°, is readily soluble in alcohol and hot benzene, sparingly in ether and cold benzene, and insoluble in water.

A mixture of *propyleneoxamide*, $C_3H_6<\begin{matrix}NH\cdot CO\\NH\cdot CO\end{matrix}>$, and *propyleneoxamic acid*, $NH_2\cdot C_3H_6\cdot NH\cdot CO\cdot COOH$, is obtained when the diamine is mixed with methyl oxalate in alcoholic solution. When the mixture is boiled for a long time with water, the amide is completely converted into the acid. The latter is a white, seemingly amorphous substance, almost insoluble in ordinary solvents, but rather less sparingly soluble in alcohol than the amide.

Propylenesuccinimide, $C_2H_4<\begin{matrix}CO\\CO\end{matrix}>N\cdot C_3H_6\cdot N<\begin{matrix}CO\\CO\end{matrix}>C_2H_4$, is obtained when the diamine (1 mol.) is heated with succinic anhydride (1 mol.) at 150—160°. It crystallises from benzene, melts at 98—100°, and is decomposed by boiling baryta water or warm sulphuric acid.

Dibenzylidenepropylenediamine, $C_3H_6(N{:}CHPh)_2$, prepared by mixing the diamine with benzaldehyde, is a yellowish oil which cannot be distilled; it is miscible with alcohol, ether, and benzene in all proportions, but is insoluble in water, and is decomposed by mineral acids. Acetophenone and the diamine react at 200°, yielding a red oil, $C_3H_6(N{:}CMePh)_2$, which distils at 350—370°.

Ethyl propylene-β-amido-α-crotonate, $C_3H_6(NH\cdot CMe{:}CH\cdot COOEt)_2$, obtained by the action of ethyl acetoacetate on propylenediamine, is a brownish oil. It is readily soluble in ether, alcohol, and benzene, but insoluble in water, and is decomposed by mineral acids.

A condensation product, probably $\begin{matrix}C_6H_4\cdot C\cdot N\cdot CMe\\|\quad\|\quad|\quad\|\\C_6H_4\cdot C\cdot N\cdot CH\end{matrix}$, is formed when aqueous propylenediamine is added to a boiling alcoholic solution of phenanthraquinone. It crystallises from alcohol in white, flexible needles, melts at 127—128°, is readily soluble in benzene, chloroform, and hot alcohol, moderately soluble in cold alcohol and ether, and insoluble in water. The *hydrochloride* crystallises in needles, and is decomposed by water and alcohol or when exposed to the air. The base separates unchanged from hot concentrated nitric acid, and is precipitated unchanged when water is added to a concentrated sulphuric acid solution. It is decomposed when heated at 200° with concentrated sulphuric acid, and is partially oxidised by chromic acid in acetic acid solution. The *platinochloride*, $(C_{17}H_{12}N_2)_2,H_2PtCl_6$, is unstable, very hygroscopic, and decomposes when heated at 200° without melting.

The compound $CPh<\begin{matrix}CPh{:}N\\N\cdot CH_2\end{matrix}>CHMe$ is the condensation product

of the diamine and benzil. It crystallises in slender needles, melts at 111—112°, and is readily soluble in benzene, chloroform, and hot alcohol, moderately soluble in cold alcohol and ether, rather sparingly in light petroleum, and insoluble in water. It is decomposed by acids.

The compound $2C_6H_{14}N_2 + H_2O$ is obtained as a bye-product in the preparation of trimethylenediamine from the bromide. It crystallises in brownish-yellow, deliquescent needles, melts below 25°, and boils considerably above 350°. It has a somewhat ammoniacal smell, and is readily soluble in all solvents. The hydrochloride and the sulphate are white, crystalline, deliquescent salts.

Diacetyltrimethylenediamine, $C_3H_6(NHAc)_2$, prepared by gradually adding acetic anhydride to the diamine and boiling the mixture, crystallises from alcohol in slightly coloured, concentrically grouped needles, melts at 79°, is readily soluble in water, alcohol, and chloroform, moderately so in benzene, but insoluble in ether and light petroleum.

Dibenzoyltrimethylenediamine, $C_3H_6(NHBz)_2$, prepared in like manner from the diamine and benzoic chloride, separates from benzene in the form of a white, crystalline powder, melts at 147—149°, is readily soluble in alcohol and chloroform, moderately so in hot benzene, and insoluble in water.

Trimethyleneoxamide, $C_3H_6(NH)_2:C_2O_2$, is obtained mixed with the amic acid when the diamine is treated with methyl oxalate in alcoholic solution. It is a white, amorphous powder, does not melt at 250°, is very sparingly soluble in boiling water, and insoluble in other solvents.

Trimethylenediamine condenses with benzaldehyde, forming a compound, $C_3H_6(N:CHPh)_2$, and with acetophenone yielding the compound $C_3H_6(N:CPhMe)_2$. Both products are oils which cannot be distilled, and are insoluble in water, but readily soluble in other solvents.

The condensation product of the diamine with phenanthraquinone is a yellow, crystalline powder, which has the composition $C_{26}H_{17}NO_2$. It is insoluble in water, sparingly soluble in alcohol and ether, moderately in benzene, and does not melt when heated at 250°.

The compound $CH_2<\begin{matrix}CH_2\cdot N:CPh\\CH_2\cdot N:CPh\end{matrix}>$ is formed when benzil and the diamine are heated together at 110° with a little alcohol. It is a reddish-brown, vitreous substance, which softens at 72°, melts completely at 80°, and is readily soluble in ordinary solvents.

F. S. K.

Septdecylamine. By G. S. Turpin (*Ber.*, **21**, 2486—2492).—Septdecylamine is best prepared by distilling septdecylstearylcarbamide with lime; stearone is formed in the reaction. The product is dissolved in alcohol, filtered from undissolved stearone, and evaporated with hydrochloric acid. The crystalline hydrochloride which separates is washed with pure ether, dissolved in a little hot alcohol, and precipitated with ether. The free base melts at 49°, boils at about 335—340°, dissolves in alcohol and ether, and readily attracts moisture and carbonic anhydride from the air. The hydrochloride crystallises from alcohol in plates of a fatty lustre. The

benzoyl-derivative, $NHBz{\cdot}C_{17}H_{35}$, forms plates of a fatty lustre and melts at 91°.

Septdecylamine septdecyldithiocarbamate, $C_{35}H_{74}N_2S_2$, prepared by mixing an ethereal solution of the amine with carbon bisulphide, is a lustrous powder melting at 90° with decomposition. When boiled with alcohol, *diseptdecylthiocarbamide*, $C_{35}H_{72}N_2S$, melting at 94° is formed.

Septdecylthiocarbimide, $C_{18}H_{35}NS$, is formed together with a small amount of diseptdecylthiocarbamide when the amine is heated with alcohol and carbon bisulphide on a water-bath. It melts at 32°, dissolves readily in alcohol and ether, and does not distil without decomposition. When heated with alcoholic ammonia at 100°, *monoseptdecylthiocarbamide*, melting at 110—111°, is formed. *Phenylseptdecylthiocarbamide*, $C_{24}H_{42}N_2S$, obtained by boiling the isothiocyanate with alcohol and aniline, melts at 79°.

Diseptdecylcarbamide, $C_{35}H_{72}N_2O$, prepared by treating the corresponding thiocarbamide with mercuric oxide, is sparingly soluble and melts at 75°.

Monoseptdecylcarbamide, $C_{18}H_{38}N_2O$, is obtained by evaporating the alcoholic solution of septdecylamine hydrochloride and potassium cyanate, and extracting the residue with absolute alcohol; it melts at 109°.

The *isocyanate* is formed as an oil when the hydrochloride is heated with a solution of carbonyl chloride in benzene at 100°. When boiled with absolute alcohol, *septdecylurethane*, $C_{20}H_{41}NO_2$, is obtained. This crystallises in lustrous plates and melts at 62°.

Phenylseptdecylcarbamide, $C_{24}H_{42}N_2O$, obtained by evaporating the thiocarbimide with a solution of aniline in benzene, crystallises in slender, lustrous needles melting at 99°. N. H. M.

Condensation of Glyoxal with Ethyl Acetoacetate. By M. Polonowsky (*Ber.*, **21**, 2499—2500).—An answer to Fittig and Schloesser (this vol., p. 1089).

Sulphacetic Acid and its Derivatives. By A. P. N. Franchimont (*Rec. Trav. Chim.*, **7**, 25—33).—Sulphopropionic acid and sulphobutyric acid can be readily prepared by the same method as that employed in the case of sulphacetic acid (compare Franchimont, *Compt. rend.*, **92**, 1054). Sulphacetic acid melts at about 75°, loses its water at about 160°, and at about 230° carbonic anhydride (about 1 mol. from 2 mols. acid) is evolved and acetic acid distils, but sulphurous anhydride and carbon are always produced in small quantities. Sulphacetic acid does not yield a nitro-derivative when the free acid or the anhydrous barium salt is heated with nitric acid. *Calcium sulphacetate* dried at 240° contains 1 mol. H_2O. The silver salt crystallises in shining plates or in prisms.

Diethyl sulphacetate can be obtained by treating the finely divided silver salt with ethyl iodide. It is sparingly soluble in water and boils at a high temperature, but undergoes decomposition. At the same time a crystalline salt, $C_6H_{14}Ag_2S_3O_{16}$, is formed in the preparation of diethyl sulphacetate by the above method, and remains when

the reaction product is extracted with ether. It dissolves in water, forming a strongly acid solution from which the normal salt crystallises. The normal salt is also formed when the acid salt is kept on blotting paper in a moist atmosphere.

Sulphobutyric acid is produced in small quantity, together with other compounds, when ethyl sulphacetate is treated with alcoholic sodic ethoxide and ethyl iodide. F. S. K.

Action of Sodium on Ethyl Butyrate and Isobutyrate. By R. Brüggemann (*Annalen*, **246**, 129—160).—The products of the action of sodium on the ethyl salt of normal butyric acid are sodium ethylenebutylenebutyrate, $C_3H_3(C_2H_4)(C_4H_8)\cdot COONa$, ethylbutyric acid, ethylenebutyric acid, ethylenebutylenedibutyric acid, $C_{14}H_{24}O_4$, dibutylenedibutyric acid, $C_{16}H_{28}O_4$, ethylenedibutylenedipropyl ketone, $C_{17}H_{28}O$, and small quantities of dipropyl ketone, ethylenedipropyl, and ethylenebutylenedipropyl ketones. No acid containing three oxygen-atoms is formed.

The chief products of the action of sodium on ethyl isobutyrate are isobutylisobutyric acid and di-isobutyrone. Ethylisobutyric, ethyleneisobutyric, and isobutylene-isobutyric acids are probably formed in small quantities. *Isobutylisobutyric* acid melts at 18° and boils at 215°. *Di-isobutyrone*, $C_3H_7\cdot CO\cdot C_3H_5{:}C_3H_5\cdot CO\cdot C_3H_7$, boils at 264—268°.

Wohlbrück (*Ber.*, **20**, 2332) states that the ethylic salt of isobutyroisobutyric acid, $C_8H_{14}O_3$, and the sodium salt of hydroxycaprylic acid, $C_8H_{16}O_3$, are formed by the action of sodium on ethyl isobutyrate mixed with ether. The author proves that the products of the reaction are the ethylic salts of isobutylisobutyric and ethylisobutyric acids, the sodium salts of isobutylene-isobutyric and ethylene-isobutyric acids and ketones. W. C. W.

Alloisomerism in the Crotonic Acid Series. By A. Michael and H. Pendleton (*J. pr. Chem.* [2], **38**, 1—5).—Compare Michael and Norton, *Amer. Chem. J.*, **2**, 15; Erlenmeyer and Müller, *Ber.*, **15**, 49.

When α-bromocrotonic acid, melting at 92°, is heated at 130—140° in sealed tubes for 15 hours, it undergoes intramolecular change, and is wholly converted into bromocrotonic acid melting at 106°. By treating this acid (m. p. 106°) with bromine in carbon bisulphide solution, it is converted into an α-β-tribromobutyric acid, melting at 115·5—116°, and identical with that obtained from allo-α-bromocrotonic acid. The yield is theoretical. *Barium α-β-tribromobutyrate* crystallises with 1 mol. H_2O and is readily soluble in water. α-β-Tribromobutyric acid loses hydrogen bromide when treated with cold alcoholic potash and is converted into α-β-dibromocrotonic acid, melting at 94°, and identical with the acid obtained by Pinner (*Ber.*, **14**, 1081) by brominating tetrolic acid. The *potassium* salt of this acid is anhydrous and readily soluble in water. The *barium* salt crystallises in prisms with $3\frac{1}{2}$ mols. H_2O, and is readily soluble in water.

These results prove that the bromine is in the α-position in both monobromocrotonic acids.

Sarnow (*Ann.*, **164**, 197) has shown that solid crotonic acid is

obtained when α-chlorocrotonic acid, melting at 97°, is reduced. The corresponding bromocrotonic acid (m. p. 106°) yields solid crotonic acid when treated with sodium amalgam, but the bromocrotonic acid melting at 92° also gives the solid acid when treated similarly, and under varying conditions so as to exclude the possibility of any secondary reaction taking place. The yield is theoretical.

It seems, therefore, that both acids are derived from solid crotonic acid.

The monobromocrotonic acids behave very differently when warmed with dilute potash; the acid melting at 106° loses 1 mol. HBr in a very short time, whereas, that melting at 92° only loses a small quantity of hydrogen bromide in the same time, and even after heating for several hours the change is not complete.

α-Chlorocrotonic acid, melting at 67°, is converted into the alloisomeric α-chlorocrotonic acid, melting at 97°, when heated for 12 hours at 150—160°, and bromomaleic acid is converted into bromofumaric acid when treated in like manner.

The results of these experiments may be summarised as follows:—When two unsaturated monobromo-acids are formed from one α-β-dibromo-acid, they are alloisomeric. If two alloisomeric α-halogen acids exist, the one with the lower melting point can be converted into the acid of higher melting point by heating. The acid of higher melting point loses hydrogen bromide more readily than that of lower melting point. F. S. K.

Action of Hypochlorous Acid on Angelic Acid. By P. Melikoff (*J. Russ. Chem. Soc.*, 1887, 524—529).—In a former paper, the author studied the action of hypochlorous acid on isocrotonic acid and obtained two chlorisocrotonic acids. In order to find out the laws governing the interesting phenomena observed in the case of isocrotonic acid, its higher homologue, angelic acid (melting point 45°), was subjected to the action of aqueous hypochlorous acid. The product of the reaction was extracted with ether, and after evaporation of the latter saturated with zinc carbonate. In this way a mixture of the zinc salts of two isomeric additive products—chlorhydroxyvaleric acids—were obtained, but as these differ in solubility, the separation of the two acids was easily effected.

(*a.*) *The less soluble zinc salt*, on decomposition with sulphuric acid and extraction with ether, yields a hydroxyvaleric acid crystallising in long, thin prisms, easily soluble in water, ether, and alcohol. It melts at 75°. Its formula is that of *β-chloro-α-methyl-α-hydroxybutyric acid*, $CHClMe{\cdot}CMe(OH){\cdot}COOH$, with which, comparison shows it to be identical. Its zinc salt, $(C_5H_8ClO_3)_2Zn$, forms fine threads consisting of prisms or plates. The calcium salt has an analogous composition. By the action of alcoholic potash on the acid, it is converted into the potassium salt of the corresponding glycidic acid, $C_5H_7KO_3 + \frac{1}{2}H_2O$; this crystallises in glistening prisms. The corresponding silver salt, $C_5H_7AgO_3$, forms thin scales. The free acid, *α-β-dimethylglycidic acid*, $C_5H_8O_3$, crystallises in microscopic prisms and smells like butyric acid. It is easily soluble in water, alcohol, and ether, and melts at 61—62°.

(*b*.) *The more soluble zinc salt* is so soluble in water that it can only be separated in the solid state by evaporation of its aqueous solution to the consistency of a syrup, and allowing it to stand for a long time, when it becomes converted into an amorphous solid. The acid, $C_5H_9ClO_3$, prepared like its isomeride, crystallises in quadratic prisms combined with a sphenoïd, easily soluble in water, alcohol, and ether, and melting at 105°. The calcium, silver, and zinc salts have the same empirical formulæ as those of the acid (*a*). The potassium salt, $C_5H_8ClKO_3$, does not undergo any change, even if its alcoholic solution is boiled in the presence of a small excess of potash; in this it differs from the salt of α-chlor-α-methyl-β-hydroxybutyric acid, obtained from tiglic acid, which under these conditions is easily decomposed with separation of potassium chloride. Another difference between the two acids is in the properties of their silver salts as well as of the free acids themselves, the α-chloro-α-methyl-β-hydroxybutyric acid melting at 62°. The corresponding glycidic acid could not be obtained. The author draws the following general conclusions: (1.) When hypochlorous acid unites with those unsaturated acids which are capable of an isomeric conversion, that is, of conversion from a less stable to a more stable form, two isomeric chlorhydroxy-acids are formed. (2.) One of these chlorinated acids is a derivative of the more stable form of unsaturated acid and identical with those chlorhydroxy-acids which are obtained by the action of hydrogen chloride on the glycidic acids corresponding with the stable forms (normal crotonic and tiglic acids). (3.) The second chlorhydroxy-acid is a derivative of the less stable form of unsaturated acid. B. B.

Synthesis of Ketonic Acids. By W. Wislicenus (*Annalen*, **246**, 306—329).—*Ethyl sodoxalacetate*, $COOEt{\cdot}CO{\cdot}CHNa{\cdot}COOEt$ (Abstr., 1887, 234, 587, this vol., p. 361), is prepared by the action of sodium on sodium ethoxide (free from alcohol) on an ethereal solution of ethyl oxalate and acetate. The product is purified by recrystallisation from absolute alcohol. It is insoluble in ether. *Ethyl oxalacetate*, $COOEt{\cdot}CO{\cdot}CH_2{\cdot}COOEt$, obtained by decomposing the sodium-derivatives with dilute sulphuric acid, is a colourless oil boiling at 131—132° under 24 mm. pressure. It is miscible in all proportions with alcohol and ether. The alcoholic solution gives a dark-red coloration with ferric chloride. Ethyl oxalacetate dissolves in a dilute solution of sodium hydroxide, and this sodium salt produces precipitates in solutions of metallic salts. The barium, calcium, lead, and silver compounds are amorphous. The zinc, cobalt, nickel, magnesium, and copper compounds are deposited from alcoholic solutions in crystals. *Ethyl phenylhydrazineoxalacetate*,

$$COOEt{\cdot}C(N_2HPh){\cdot}CH_2{\cdot}COOEt,$$

crystallises in quadratic plates. It is freely soluble in alcohol and ether, and melts at 76—78°, but decomposes on exposure to a temperature of 100°, yielding ethyl alcohol and *ethylic phenylpyrazolone-carboxylate*, $PhN\langle\begin{matrix}CO\text{——}CH_2\\N{:}C(COOEt)\end{matrix}\rangle$.

The pyrazolone-derivative melts at 180—182° and dissolves in

alcohol. It yields a yellow isonitro-compound. The free acid forms a red isonitroso-derivative. W. C. W.

Ethylic Methyloxalacetate and Ethyloxalacetate. By E. Arnold (*Annalen*, **246**, 329—338).—The preparation and some of the properties of ethylic methyloxalacetate have been previously described (this vol., p. 361). The phenylhydrazine-derivative crystallises in small plates, and melts at 99—100°; at 120° it decomposes, yielding the ethyl salt of 1 : 4 : 5 : 3 phenylmethylpyrazolonecarboxylic acid. The free acid is deposited from alcohol in plates, and melts at 220—221°. The ethyl salt forms needle-shaped crystals. It melts at 148—149°, and dissolves freely in ether, chloroform, and benzene.

Ethylic ethyloxalacetate, COOEt·CO·CHEt·COOEt, is prepared by the action of sodium ethoxide (free from alcohol) on a mixture of ethyl oxalate and butyrate. It is a liquid boiling at 136—138° under 20 mm. pressure. Ferric chloride produces a red coloration. Ethylic ethyloxalacetate is decomposed by boiling with dilute sulphuric acid, yielding butyrylformic acid. W. C. W.

Action of Ethyl Iodide and Zinc on Ethyl Malonate. By S. Schukoffsky (*J. Russ. Chem. Soc.*, 1887, 601—604).—When a mixture of ethyl malonate with ethyl iodide is heated with zinc, using a reflux condenser, large quantities of a gas are given off which is not absorbed by bromine. The principal product of the reaction is the ethyl salt of diethylmalonic acid, $CEt_2(COOEt)_2$, a liquid boiling at 220°, and having the sp. gr. 1·0071 at 0°, and 0·9901 at 15°. The free diethylmalonic acid was obtained by hydrolysis of the ethyl salt with alcoholic potash, and the calcium, sodium, potassium, and silver salts were prepared. When heated to 170—180°, diethylmalonic acid is decomposed into carbonic anhydride and diethylacetic acid.

B. B.

Oxytetric and Hydroxytetric Acids. By A. Gorboff (*J. Russ. Chem. Soc.*, 1887, 605—615).—Demarçay's tetric acid and its homologues of the supposed general formula $C_{3(m+3)}H_{6(m+1)+2}O_7$, have, according to W. Pavloff's investigation, the general formula $C_nH_{2n-4}O_3$, and his oxytetric series of the formula $C_{6(m+3)}H_{3(m+1)+2}O_{10}$, most probably (as pointed out by Beilstein) the formula $C_nH_{2n-4}O_4$, which, as the author shows, although at first sight probable, is not in accordance with Demarçay's analytical results. The author has compared at length the physical and chemical properties of oxytetric acid, its derivatives, and salts with those of *mesaconic acid*, $C_3H_4(COOH)_2$, and finds a striking agreement, which leads him to believe in their identity. The *oxypentic acid* would then be identical with hydromucic acid, the *oxyhexic acid* with terebic acid, and the *hydroxytetric acid* with pyrotartaric acid, as shown in a table comparing them. The *hydroxypentic acid* is ethyltartaric acid, the *hydroxyhexic acid* propylsuccinic acid, and the *hydroxyisohexic acid* is isopropyltartaric acid. B. B.

Derivatives of Carbamide. By A. P. N. Franchimont and E. A. Klobbie (*Rec. Trav. Chim.*, **7**, 12—24).—*Nitrohydantoïn*,

$$NH<\begin{matrix}CO\cdot NH\text{———}\\CO\cdot CH(NO_2)\end{matrix}>,$$

prepared by evaporating a solution of hydantoïn with five times its weight of nitric acid, crystallises from alcohol in short prisms, and from water in small, shining needles melting at about 170° with decomposition (compare Franchimont, this vol., pp. 1052 and 1064).

Nitrolactylcarbamide, $NH<\begin{matrix}CO\text{———}NH\\CO\cdot CMe(NO_2)\end{matrix}>$, prepared in like manner, crystallises from water and alcohol in rhombic plates, melts at about 148° with decomposition, and is readily soluble in water, rather less so in alcohol, and almost insoluble in ether, benzene, and chloroform. It is decomposed when boiled with water.

α-Ureidopropionamide, $CONH_2\cdot NH\cdot CHMe\cdot CONH_2$, was obtained in the preparation of lactylcarbamide by Heintz's method. It crystallises from alcohol in small, slender needles, melts at 196°, and is soluble in water, but insoluble in ether. When heated with hydrochloric acid, it yields lactylcarbamide and ammonium chloride.

α-Ureidopropionitrile, $CONH_2\cdot NH\cdot CHMe\cdot CN$, also obtained in the preparation of lactylcarbamide, crystallises in small prisms, melts at 106°, and is very readily soluble in water and alcohol, but only very sparingly in ether. When heated with hydrochloric acid, it yields lactylcarbamide and ammonium chloride.

Dinitroethylenecarbamide, $CO<\begin{matrix}NH\cdot CH(NO_2)\\NH\cdot CH(NO_2)\end{matrix}>$, obtained by treating ethylenecarbamide with five times its weight of nitric acid, crystallises in colourless prisms, melts at 210° with decomposition, and is sparingly soluble in alcohol and water. When it is boiled with water, carbonic anhydride is evolved, the solution becomes strongly acid, and on evaporation a crystalline compound is obtained. This product has the composition of a dinitroethylenediamine; it melts at about 174° with decomposition, is soluble in water and alcohol, and is immediately decomposed by concentrated sulphuric acid or nitric acid with evolution of nitrous oxide (2 mols.). The *potassium* salt crystallises in colourless needles, melts at 210°, and is insoluble in absolute, but soluble in dilute, alcohol.

Dinitroglycolurile, $CO<\begin{matrix}NH\cdot C(NO_2)\cdot NH\\|\\NH\cdot C(NO_2)\cdot NH\end{matrix}>CO$, prepared by nitrating glycolurile, is almost insoluble in boiling water or alcohol, and decomposes at about 217° without melting. When boiled with water, it is decomposed into carbonic anhydride (1 mol.), nitrous oxide (2 mols.), and a colourless, crystalline compound which is insoluble in most ordinary solvents.

Glycoldimethylurile,

$$CO<\begin{matrix}NMe\cdot CH\cdot NMe\\|\\NH\cdot CH\cdot NH\end{matrix}>CO \text{ or } CO<\begin{matrix}NMe\cdot CH\cdot NH\\|\\NH\cdot CH\cdot Me\end{matrix}>CO,$$

prepared by treating an aqueous solution of a mixture of glyoxal and methylcarbamide with hydrochloric acid, crystallises from boiling

water in slender needles, begins to melt at 210°, and fuses completely at 260°. It is readily soluble in boiling water, sparingly in alcohol, and insoluble in ether and light petroleum.

Dinitroglycoldimethylurile, $C_6H_8N_6O_6$, is a white powder, insoluble in boiling water or alcohol, and is not decomposed when boiled with water.

Isosuccinic-ureïd, $CHMe<\begin{matrix}CO\cdot NH\\CO\cdot NH\end{matrix}>CO$, obtained by heating a mixture of isosuccinic acid and carbamide with phosphorus oxychloride, sublimes at about 190° in a partial vacuum, forming long, colourless, shining plates, melts at 192°, and is readily soluble in water and alcohol, but only sparingly in ether. When treated with nitric acid, it yields a compound which crystallises in needles and is readily soluble in water, ethyl alcohol, methyl alcohol, and ether, but only sparingly in chloroform and benzene, and insoluble in light petroleum. Its composition is $C_{20}H_{22}N_{12}O_{23}$. F. S. K.

Constitution of Benzene. By A. Ladenburg (*Annalen*, **246**, 382—384).—The author maintains that Baeyer's proposed formula for benzene is identical with Claus's diagonal formula for benzene. So long as we regard the carbon-atom as having four equal affinities, Claus's formula cannot be reconciled with the existence of three isomeric benzene substitution products. W. C. W.

Configuration of the Benzene Molecule. By H. Sachse (*Ber.*, **21**, 2530—2538).—The arrangement ascribed to the benzene molecule is shown by a model constructed as follows:—Two parallel triangles are removed from a cardboard model of an octahedron and a regular tetrahedron placed on each of the remaining six triangles, so that the latter form the bases of the tetrahedra. These represent the six carbon-atoms, whilst the hydrogen-atoms are supposed to be situated at the free angles. The ring thus formed of six carbon-atoms is more stable than a ring formed of any other number of carbon-atoms, as each atom is so attached to the two next atoms that it is in equilibrium, and as a periodic movement of single carbon-atoms is not possible, the system can only move as a whole.

The constitution of naphthalene and pyrene based on the above benzene formula is also discussed. N. H. M.

Dihydroxyquinone and Tetrahydroxybenzene. By R. Nietzki and F. Schmidt (*Ber.*, **21**, 2374—2379).—*Dihydroxyquinone*,

$$C_6H_2(OH)_2O_2$$

(symmetrical), is formed when the diamidoresorcinolimide, prepared by Typke's method, is dissolved in 10 parts of 10 per cent. potash or soda, and the solution kept at 70° until, on adding hydrochloric acid to a small portion, straw-coloured needles are precipitated. Alkali is then added, and the salt which separates from the cold solution dissolved in water, and decomposed by an acid. The quinone is readily soluble in alcohol, glacial acetic acid, and ethyl acetate, crystallising from the last-named solvent in dark-yellow, indented needles which

have a bluish tinge; it is almost insoluble in cold, and only sparingly soluble in hot water, and is decomposed when the solution is boiled. It sublimes rather readily without melting. When treated with fuming nitric acid, it yields nitranilic acid. The *sodium*-derivative, $C_6H_2(ONa)_2O_2$, and the potassium-derivative are readily soluble in water, yielding red solutions from which they crystallise in long, dark-red needles; they are sparingly soluble in alkalis and alcohol, and when precipitated by these reagents and dried are obtained in the form of an orange-yellow powder. The *barium*-derivative,

$$C_6H_2BaO_4 + H_2O,$$

is sparingly soluble in water, and crystallises in almost black needles with a bluish reflex. The *silver*-derivative is amorphous, and when treated with methyl alcohol and methyl iodide yields the *methyl*- and *dimethyl*-derivatives. The latter crystallises in bright yellow plates, and can be separated from the former by means of ammonia, in which it is soluble. It decomposes when heated to 220°, does not give a red coloration with concentrated sulphuric acid, and when treated with bromine yields a colourless, viscous substance. The *oxime*, $C_6H_2(OH)(NOH)_2$, crystallises in colourless plates and dissolves in alkalis, forming a brown solution from which it is precipitated unchanged by acids.

Tetrahydroxybenzene, $C_6H_2(OH)_4$ [$H_2 = 1 : 4$], prepared by warming the preceding compound with stannous chloride, is very readily soluble in water, alcohol, and ether, moderately soluble in hydrochloric acid or glacial acetic acid, and crystallises from the last-named solvent in greyish plates melting at 215—220°. Aqueous solutions quickly become brown on exposure to the air, and alkaline solutions are soon converted into solutions of the corresponding salt of the dihydroxyquinone. Ferric chloride precipitates the dihydroxyquinone from aqueous solutions of the tetrahydroxy-compound. The *acetyl*-derivative, $C_6H_2(OAc)_4$, crystallises from glacial acetic acid in small, colourless, rhombic plates melting at 217°.

The dihydroxyquinone and tetrahydroxybenzene described above are not identical with the compounds described under the same names by Loewy (Abstr., 1886, 1028). F. S. K.

Dichroïns obtained by the Action of Aqua Regia and Bromine Aqua Regia. By H. Brunner and P. Chuit (*Ber.*, **21**, 2479—2486).—*Monochloro-α-pentaresorcinoldichroïn ether*, $C_{66}H_{48}ClN_3O_{18}$, is obtained by heating 15 grams of resorcinol, 60 grams of water, 20 c.c. of nitric acid (sp. gr. = 1·39), and 60 c.c. of hydrochloric acid (sp. gr. = 1·2) for half an hour on a water-bath. It is a dark violet substance, of metallic lustre, insoluble in chloroform, benzene, and carbon bisulphide, soluble in alcohol and ether; the alkaline solution is of a pure blue colour, with a brown fluorescence. The *acetyl-derivative*, $C_{66}H_{40}Ac_8ClN_3O_{18}$, prepared by heating the dye with 3 parts of acetic anhydride and 2 parts of dry sodium acetate at 120° for two hours, forms a red, amorphous mass, sparingly soluble in water, soluble in alcohol, chloroform, and benzene.

Monobromo-α-tetraresorcinoldichroïn ether, $C_{48}H_{35}BrN_2O_{12}$, is formed

when 15 grams of resorcinol, 70 c.c. of water, 60 c.c. of hydrobromic acid (sp. gr. = 1·49), and 20 c.c. of nitric acid (sp. gr. = 1·39), are heated in a large flask on a water-bath. The product is washed with water, dried, extracted with benzene, which removes a brown substance, and dissolved in ether. It is a brownish-violet substance of metallic lustre, readily soluble in alcohol and acetone, almost insoluble in chloroform and benzene. The *acetyl-derivative*, $C_{48}H_{29}Ac_6BrN_2O_{13}$, is an orange-red, amorphous powder, melts at 120°, and dissolves readily in hot alcohol. When the pure dye is heated at 120°, the *anhydride*, $C_{48}H_{33}BrN_2O_{12}$, is formed. This dissolves in alkalis, with violet colour and brown fluorescence. The sparingly soluble dichroïn has the formula $C_{36}H_{27}BrN_2O_{11}$.

Monochloro-α-orcinoldichroïn, $C_{21}H_{20}ClNO_6$, was prepared by Reymann (*Ber.*, **13**, 809), who ascribed to it the formula $C_{21}H_{17}ClN_2O_6$. It is best prepared by heating 10 grams of orcinol, 10 c.c. of nitric acid, and 30 c.c. of hydrochloric acid on a water-bath, washing the product with water, dissolving in ammonia or soda, and precipitating with acid. When dried, it acquires a greenish colour and metallic lustre; it is soluble in alcohol, ether, and acetone, insoluble in benzene and chloroform. The *acetyl compound*, $C_{21}H_{16}Ac_4ClNO_6$, dissolves readily in benzene and chloroform, sparingly in alcohol.

When orcinol (4 grams) dissolved in a little hot water is heated on a water-bath with nitric acid (sp. gr. = 1·39, 10 c.c.), and hydrobromic acid (sp. gr. = 1·49, 30 c.c.), a red substance of the formula $C_{48}H_{27}Me_6BrN_2O_3$ is obtained. This dissolves in alkalis, yielding a peach-coloured solution with a red fluorescence. *Monobrom-α-orcinoldichroïn*, $C_{21}H_{20}BrNO_6$, is also formed in the reaction. It is a brown powder, soluble in alcohol and hot water, almost insoluble in ether and benzene; the solution in alkalis is reddish-violet, with a brown fluorescence.

Aqua regia acts on phenol with formation of a red dye, which dissolves in alkalis with deep blue colour. When hydrobromic acid is used, a crystalline compound resembling dibromorthonitrophenol is obtained. It melts at 115°; the lead, barium, potassium, sodium, and silver salts are respectively yellowish-red, orange-yellow, red-yellow, and red. Dichroïns are therefore only formed when the nitrogen-atom is in the para-position. The dichroïns obtained from resorcinol and orcinol belong to the α-series, and contain the group $C_6{\cdot}N(O{\cdot}C_6)_2$. N. H. M.

Decomposition Products of Panicole. By G. Kassner (*Arch. Pharm.* [2], **26**, 536—541).—Panicole, obtained by the author from the fatty oil of millet as the crystallised constituent of that oil, (*Arch. Pharm.* [3], **25**, 395), was considered most probably to be a mixed ether, and not an alcohol. Under the action of hydriodic acid in a sealed tube at 150°, methyl iodide was produced. The formula of panicole being $C_{13}H_{20}O$, and methyl iodide being obtained from it, as stated above, the compound must be a mixed ether, and contain a methoxyl-group. The reaction with hydriodic acid was not perfectly satisfactory, but with fuming hydrochloric acid, and some chloroform to increase the surfaces in contact, the decomposition was complete

after heating at 160° in a sealed tube for several hours. Methyl chloride was obtained, and also a solid which melted at 78° (panicole melts at 285°), but could not be obtained in a crystalline form; its composition was $C_{12}H_{17}\cdot OH$. Hence panicole has the formula $C_{12}H_{17}\cdot OCH_3$. The substance, $C_{12}H_{17}\cdot OH$, is a tertiary alcohol, a phenol. Concentrated sulphuric acid turns it raspberry-red, and dissolves it; on pouring this solution into much water, a light flocculent precipitate is thrown down, probably as a sulphonic acid. A complete crystallographic description of panicole is given. J. T.

Reduction-products of Metadinitroparacetotoluide. By Z. Bankiewicz (*Ber.*, **21**, 2402—2411).—*Nitroethenyldiamidotoluene*,

$$NO_2\cdot C_6H_2Me\langle\begin{matrix}-N\\NH\end{matrix}\rangle CMe,$$

prepared by treating metadinitracetotoluide with a slight excess of alcoholic ammonium sulphide, crystallises from dilute alcohol or hot water in slender needles, melts at 246°, is very readily soluble in alcohol, sparingly in ether, and almost insoluble in cold water or soda. It yields ethenyltriamidotoluene when reduced with tin and hydrochloric acid (compare Niementowski, Abstr., 1886, 544). The *hydrochloride*, $C_9H_9N_3O_2,HCl$, crystallises from water in large, well-defined prisms, and is readily soluble in alcohol. The *sulphate* is very readily soluble in alcohol and alcoholic ether, and crystallises in long, colourless needles. The *nitrate* crystallises in yellow, transparent plates, and melts at 207°; it is moderately soluble in cold, and readily in hot water.

When dinitracetotoluide is treated with alcoholic ammonium sulphide, in quantity insufficient for complete reduction, and the reduction-product mixed with soda, nitroethenyldiamidotoluene and hydrazodinitracetotoluide remain, and the solution contains three compounds, which are precipitated on adding acetic acid. (1.) An azoxy-compound, which is very sparingly soluble in hot alcohol, and melts at 236°. (2.) An azo-compound, which is readily soluble in hot alcohol, and crystallises in brown needles melting at 242—244°. (3.) Nitroxyethenyldiamidotoluene, which is formed when the reduction-product is boiled with water or dilute acids.

Nitroxyethenyldiamidotoluene, $NO_2\cdot C_6H_2Me\langle\begin{matrix}NH\cdot CMe\\|\\N\end{matrix}\rangle O$, crystallises in shining, green needles containing water, melts at 255—256°, and is readily soluble in hot water or dilute alcohol, but only very sparingly in absolute alcohol, ether, or benzene. It dissolves in soda with a red coloration, and is also soluble in concentrated ammonia. It is not acted on at 200° by concentrated hydrochloric acid, and is not hydrolysed by alcoholic potash. When treated with nitrous acid, it yields crystalline, unstable nitroso-compounds. It yields diamidoacetotoluide (Niementowski, *loc. cit.*), when reduced with tin and hydrochloric acid. The hydrochloride is crystalline.

Dinitrodiethenyltetramidoditolyl,

$$CMe\langle\overset{NH}{N-}\rangle C_6HMe(NO_2)\cdot C_6HMe(NO_2)\langle\overset{NH}{-N}\rangle CMe,$$

is obtained when hydrazodinitracetotoluide (see above) is treated with dilute hydrochloric acid. It crystallises in long prisms or microscopic needles, melts at 242°, is readily soluble in ethyl and methyl alcohol, moderately soluble in hot water, but only sparingly in ether and soda. The *sulphate* crystallises in concentrically grouped needles, and is readily soluble in water, but only sparingly in alcohol. The *hydrochloride*, $C_{18}H_{16}N_6O_4,2HCl$, crystallises in colourless, flat needles, and sublimes at 100°. The *nitrate* crystallises from water in thick, yellowish needles melting at 213—214° with decomposition.

Diacetohexamidoditolyl, $NHAc\cdot C_6HMe(NH_2)_2\cdot C_6HMe(NH_2)_2\cdot NHAc$, prepared by reducing the preceding compound with tin and hydrochloric acid, crystallises with 5 mols. H_2O, melts at 196°, and is very readily soluble in alcohol, moderately in ether and benzene, but almost insoluble in water. It oxidises quickly in hot alcoholic solution. The *hydrochloride*, $C_{18}H_{24}O_2N_6,2HCl + 2H_2O$, crystallises in colourless needles, and is very readily soluble in water. The *picrate*,

$$C_{18}H_{24}O_2N_6,2C_6H_3N_3O_7,$$

and the *nitrate* crystallise in red needles. F. S. K.

Trinitrometaphenylenedimethyldinitramine. By P. VAN ROMBURGH (*Rec. Trav. Chim.*, **7**, 1—11).—Pure tetramethylmetaphenylenediamine melts at −2°, and boils at 266—267° (748 mm. pressure) (compare Wurster and Morley, *Ber.*, **12**, 1814). It combines with symmetrical trinitrobenzene (1 mol.), yielding a product which crystallises in long, almost black, shining needles melting at 121°; it also combines with metadinitrobenzene (1 mol.); the product crystallises in dark, garnet-red needles, which melt at 58°.

Trinitrometaphenylenedimethyldinitramine, $C_6H(NO_2)_3(NMe\cdot NO_2)_2$, is obtained by gradually adding tetramethylmetaphenylenediamine, dissolved in twice its volume of concentrated sulphuric acid to nitric acid of sp. gr. 1·48. It can be crystallised from alcoholic acetone, but is almost insoluble in most ordinary solvents, excepting acetone, and is decomposed when heated at about 205°. It yields methylamine when boiled with dilute potash.

Trinitrodimethylmetaphenylenediamine, $C_6H(NO_2)_3(NHMe)_2$, obtained by boiling the preceding compound with phenol, separates from phenol in small, yellow crystals melting at 235° with decomposition. It is sparingly soluble in acetic acid and all ordinary solvents, and less readily in acetone than the nitramine. It is reconverted into the nitramine by cold, concentrated nitric acid, and when boiled with very dilute potash it yields methylamine and trinitroresorcinol. Its constitution is probably $[(NHMe)_2 : (NO_2)_3 = 1 : 3 : 2 : 4 : 6]$.

F. S. K.

Chromogenic Carbins: Constitution of Rosaniline Salts. By V. v. RICHTER (*Ber.*, **21**, 2475—2478).—Ethyl dinitrophenylacetate is formed in almost theoretical quantity by the action of orthoparadinitrobromobenzene on the sodium-derivative of ethyl

orthoparadinitrophenylacetate. The reaction confirms the presence of a CH-group in the compound (compare this vol., p. 1189).

Paranitrobenzyl cyanide (Gabriel, *Ber.*, **14**, 2342) dissolves in alcoholic potash or soda with intense carmine-red coloration and formation of salts, $NO_2 \cdot C_6H_4 \cdot CHX \cdot CN$. Orthonitrobenzyl cyanide, on the other hand, dissolves in alcoholic alkali with intense violet-blue coloration (Bamberger, Abstr., 1887, 131).

When orthoparadinitrobenzene is added to an alcoholic solution of paranitrobenzyl cyanide and sodium ethoxide, an intensely violet-blue solution is obtained. The product, which is probably *paranitrophenyl-orthoparadinitrophenylcarbin cyanide*, $CN \cdot CH(C_6H_4 \cdot NO_2) \cdot C_6H_3(NO_2)_2$, is almost colourless, dissolves in alkalis, ammonia, and alkaline carbonates with intense dark blue coloration, and is precipitated unchanged on addition of an acid. The *sodium*-derivative is a greenish-black amorphous powder.

The name carbin-dyes is ascribed to the above compounds, as well as to the rosaniline salts, which are probably similarly constituted, carbin being the name for all methane-derivatives in which positive or negative groups are substituted for two or three hydrogen-atoms, and the remaining atom or atoms can be readily displaced by radicles of the opposite character. The acid carbins, as for instance trinitrophenylcarbin, $CH(C_6H_4 \cdot NO_2)_3$, and the carbin bases, as triamidophenylcarbin, are colourless, whilst the salts $CNa(C_6H_4 \cdot NO_2)_3$ and $CCl(C_6H_4 \cdot NH_2)_3$ are coloured. N. H. M.

Eurhodines. By O. N. Witt (*Ber.*, **21**, 2418—2421).—The author proposes to extend the name of "eurhodine" to all polyamido-derivatives of azines, and that of "saffranine" to all amido-derivatives of azonium bases. The saffranines and eurhodines are closely related to the indamines, which are very often formed as intermediate products in the preparation of these dyes. To determine whether this intermediate formation of an indamine is necessary, the author endeavoured to obtain dyes from metaxylylenediamine, [Me : $(NH_2)_2$ = 1 : 3 : 4 : 6], which he prepared by reducing metanitrometaxylydine. Metaxylylenediamine is not unstable as stated by Fittig, Ahrens, and Mattheides (*Annalen*, **147**, 18). The *hydrochloride* crystallises in white needles.

When the hydrochloride is well mixed with dry sodium acetate (1 mol.) and nitrosodimethylaniline (1 mol.), and the mixture covered with glacial acetic acid and boiled, a violent reaction takes place, but no eurhodine is formed. Metaxylylenediamine and quinonechlorimide in acetic acid solution do not yield a dye.

These results show that this diamine which cannot form an indamine is also unable to produce a eurhodine, and consequently the number of eurhodines which can be formed by this method is considerably limited.

Metaxylylenediamine does not show the usual reactions of meta-diamines; it does not yield vesuvin when treated with sodium nitrite, and when mixed with diazobenzenesulphonic acid and sodium acetate no chrysoïdinesulphonic acid is formed. It is, however, readily diazotised, and an aqueous solution of the diazo-compound forms a

brown azo-dye with metatoluylenediamine, the properties of which coincide with those of the typical toluylene-brown; it also forms a red dye with $\alpha_1\alpha_2$-naphtholsulphonic acid and a reddish-brown dye with Brönner's β-naphthylaminesulphonic acid. F. S. K.

Derivatives of Carbizin and Thiocarbizin. By M. Freund and B. B. Goldsmith (*Ber.*, **21**, 2456—2469).—The compounds previously described (this vol., p. 686; *Ber.*, **21**, 1240) are derivatives of carbizin, $CO<^{NH}_{NH}>$.

Formylphenylcarbizin, $CO<^{NPh——}_{N(CHO)}>$, prepared by heating a benzene solution of carbonyl chloride with formylphenylhydrazide, crystallises in needles, melts at 73°, boils at 255—256°, is readily soluble in alcohol, ether, chloroform, benzene, and glacial acetic acid, moderately in carbon bisulphide, and sparingly in water. It is reconverted into the hydrazide when boiled with sodium carbonate.

Acetylphenylcarbizin (*loc. cit.*) is converted into acetylphenylhydrazine when boiled with sodium carbonate; and when heated with potash it is decomposed into carbonic anhydride, acetic acid, and phenylhydrazine. It is not reduced by zinc and glacial acetic acid, but when warmed with phosphorus and hydriodic acid considerable quantities of aniline hydriodide and carbonic anhydride are formed. It is completely decomposed when boiled with sodium ethoxide, or when heated with alcoholic ammonium sulphide.

Propionylphenylhydrazine, NHPh·NH·COEt, prepared by heating propionamide with phenylhydrazine, crystallises from chloroform in plates, and melts at 157—158°.

Propionylphenylcarbizin, $CO<^{NH——}_{N(COEt)}>$, obtained by heating the hydrazine with carbonyl chloride, crystallises from alcohol in white needles, and melts at 62—63°.

Benzoylphenylcarbizin, $<^{CO-}_{NPh}>N\cdot COPh$, prepared by heating benzoylphenylhydrazide with a benzene solution of carbonyl chloride, crystallises from alcohol in white needles, and from glacial acetic acid in rhombic plates which melt at 113—114°, and are insoluble in water.

Succinylphenylhydrazine, $N_2H_2Ph\cdot CO\cdot C_2H_4\cdot CO\cdot N_2H_2Ph$, crystallises from glacial acetic acid in yellowish plates, melts at 208—209°, and is sparingly soluble in alcohol.

Succinylphenyldicarbizin, $<^{NPh}_{CO-}>N\cdot CO\cdot C_2H_4\cdot CO\cdot N<^{NPh}_{CO-}>$, melts at 225°.

Phenylcarbizincarboxylamide, $<^{CO-}_{NPh}>N\cdot CO\cdot NH_2$, is obtained when finely divided phenylsemicarbazide is mixed with a benzene solution of carbonyl chloride, the mixture kept for 8 to 10 hours, and the benzene evaporated at the ordinary temperature. It crystallises from hot alcohol in needles, melts at 166—167°, is moderately soluble in benzene and chloroform, sparingly in water, and insoluble in ammonia and alkalis.

Diphenylsemicarbazide is obtained by mixing phenylhydrazine and phenyl cyanate in ethereal solution. (Compare Skinner and Ruheman, this vol., p. 552.) It melts at 173°.

Phenylcarbizincarboxyanilide, $<^{NPh}_{CO-}>N\cdot CO\cdot NHPh$, crystallises from glacial acetic acid in needles, melts at 173°, and is soluble in alcohol, but only very sparingly soluble in water.

Phenylcarbizinthiamide, $<^{NPh}_{CO-}>N\cdot CS\cdot NH_2$, the reaction product of phenylthiosemicarbazide and carbonyl chloride, separates from glacial acetic acid in microscopic crystals, melts at 270°, is sparingly soluble in glacial acetic acid, and insoluble in most solvents except aniline.

Phenylcarbizinthianilide, $<^{NPh}_{CO-}>N\cdot CS\cdot NHPh$, crystallises from hot alcohol, carbon bisulphide or benzene in needles, melts at 188°, and is very readily soluble in chloroform, but insoluble in water.

Acetylphenylthiocarbizin, $<^{NPh}_{CS-}>NAc$, prepared by heating acetylphenylhydrazine with carbon thiochloride in benzene solution, crystallises from alcohol in prisms, melts at 73—74°, and boils at 275°. It is readily soluble in ether, chloroform, benzene, carbon bisulphide, and acetone, but insoluble in water. When boiled with mercuric oxide and alcohol, it yields acetylphenylcarbazin. It is isomeric with the compound described by Fischer (*Annalen*, **202**, 126), the constitution of which is probably $<^{NPh}_{\quad N}>CSAc$.

Benzoylphenylthiocarbizin, $<^{NPh}_{CS-}>N\cdot COPh$, prepared in like manner, crystallises from alcohol in slender, white needles, melts at 110°, is readily soluble in chloroform, ether, and benzene, moderately so in alcohol and light petroleum, but insoluble in water. It yields benzoylphenylcarbizin when boiled with alcohol and mercuric oxide.

F. S. K.

Action of Phenylhydrazine on the Alkyl Salts of Halogen Ketonic Acids and Halogen Ketones. By G. Bender (*Ber.*, **21**, 2492—2498).—*Ethyl phenylhydrazineacetylacrylate*,

$$NHPh\cdot N{:}CMe\cdot CH{:}CH\cdot COOEt,$$

is prepared by mixing solutions of ethyl bromolevulinate and phenylhydrazine in four parts of absolute alcohol, which have been previously cooled at 0°. The whole is then kept for 24 hours at the ordinary temperature. The product is filtered, the crystals dried on porous plates, powdered, washed with water, and crystallised from absolute alcohol. It forms yellow scales, melts at 117·5°, and is readily soluble in hot alcohol. When reduced with tin and hydrochloric acid, methylindoleacetic acid is formed. *Phenylhydrazineacetylacrylic acid*, $C_{11}H_{12}O_2N_2$, crystallises in splendid, dark yellow crystals which melt at 157° with effervescence.

Phenylhydrazine reacts with ethyl chlorolevulinate with formation of two compounds; the one is readily soluble and melts at 123°, the other,

$C_{14}H_{21}O_4N_2$, which is sparingly soluble, melts at 205° with evolution of gas.

Bromine acts on ethyl benzoylacetate with formation of two compounds, one of which is readily soluble, and melts at 130—132°; the other crystallises in sparingly soluble, brick-red needles melting at 165°.

When dibromacetophenone (1 mol.) is kept for two days with phenylhydrazine (4 mols.), the compound NHPh·N:CPh·CH:N·NHPh is obtained. This crystallises from alcohol in intensely yellow prisms melting at 148°.

When 5 grams of chloracetone dissolved in 5 to 6 vols. of absolute alcohol is quickly added to a solution of 18 grams of phenylhydrazine dissolved in 5 to 6 vols. of alcohol cooled at −16° to −18°, the whole soon solidifies to a yellow, crystalline mass. After being kept for ¼ hour in the freezing mixture, the crystals are dried, digested with water, and crystallised from alcohol. The new compound, which has the formula $C_{24}H_{26}N_6$, forms pale yellow crystals melting at 157—158°. When boiled with acetic anhydride, the compound $C_{26}H_{28}N_6O$ crystallising in pale yellow needles is obtained; it melts at 220°. When the compound $C_{24}H_{26}N_6$ is treated with sodium nitrite and acetic acid, a compound of the formula $C_{24}H_{22}N_6$ is formed; this crystallises in very lustrous, flat, reddish-brown needles melting at 104°.

N. H. M.

New Chromogenic Groups. By V. v. Richter (*Ber.*, **21**, 2470 2475).—The bye-product which the author obtained in the preparation of ethyl dinitrophenylacetoacetate from orthoparadinitrobromobenzene and ethyl sodacetoacetate has already been described by Heckmann (*Annalen*, **220**, 137), who assigned to it the composition $C_{24}H_{18}N_6O_{15}$. This substance is ethyl bi-dinitrophenylacetate, and has the constitution $CH[C_6H_3(NO_2)_2]_2·COOEt$. It is best prepared by treating the sodium-derivative of ethyl dinitrophenylacetoacetate with dinitrobromobenzene, acetic acid being also formed. The yield is two-thirds of the theoretical quantity, It is also obtained in small quantities when ethyl sodiomalonate (1 mol.) is treated with dinitrobromobenzene, but the principal product is ethyl dinitrophenylmalonate.

Ethyl dinitrophenylmalonate, $C_6H_3(NO_2)_2·CH(COOEt)_2$, is very readily soluble in warm alcohol or ether, and crystallises in thick, yellowish prisms, melting at 51°. It dissolves in alkalis with a red coloration and is precipitated unchanged by acids. The *sodium*-derivative, obtained by mixing the ethyl salt with concentrated soda, is insoluble in moderately concentrated alkalis, but dissolves in water with a brown coloration forming a solution from which acids precipitate the pure ethyl salt.

Ethyl bi-dinitrophenylacetate is obtained when this sodium compound is treated with dinitrobromobenzene in alcoholic solution. It is sparingly soluble in alcohol and ether, and yields a *sodium*-derivative, $C_{16}H_{11}NaN_4O_{10}$, which crystallises in golden plates and explodes when heated at about 80°, or when heated with concentrated sulphuric acid. This sodium salt is very hygroscopic and absorbs carbonic anhydride from the air. The sodium and potassium salts dissolve in

water or alcohol, forming deep-blue solutions which are decolorised on exposure to the air, but regain their colour when alkalis are added.

These experiments show that the chromogenic nature of these compounds is due to the substitution of metals for the hydrogen of the CH_2 or CH group, and also to the dinitrophenyl-group with which the carbon is combined. When the compound contains one dinitrophenyl-group, it forms brownish-red salts, whereas when two such groups are present the salts are blue.

Bi-dinitrophenylmethane, $CH_2[C_6H_3(NO_2)_2]_2$, gives deep blue solutions with alcoholic potash or soda.

These chromogenic compounds may be classed with the nitro-dyes.

F. S. K.

Decomposition Products of Chloranilic and Bromanilic Acids. By A. HANTZSCH (*Ber.*, **21**, 2421—2444).—The first product of the action of chlorine on an aqueous solution of potassium chloranilate is an additive compound, $C_6(OH)_2Cl_2O_2,Cl_2$, which cannot, however, be isolated as it is almost immediately converted into trichlorotriketopentamethylene (see below).

Trichlorodiketopentamethylenehydroxycarboxylic acid,

$$<\begin{matrix}CO\cdot CCl_2-\\CO\cdot CHCl\end{matrix}>C(OH)\cdot COOH,$$

the second product of the action of chlorine on potassium chloranilate, is obtained when a solution of pure sodium hypochlorite (1 mol.) is gradually added to a solution of pure potassium chloranilate (1 mol.), the mixture acidified with hydrochloric acid and the product extracted with ether. The yield is almost theoretical. It crystallises from water in anhydrous prisms and from ether in transparent prisms which seem to contain ether. It melts at 171° with decomposition, and is very readily soluble in ether, alcohol, and water, but almost insoluble in benzene. It has a strongly acid reaction, and forms salts, most of which are amorphous and readily soluble in water, but the *lead* salt, $C_6HPbCl_3O_4$, the *mercurous* and the unstable *silver* salt, can be obtained by direct precipitation. It instantly reduces warm ammoniacal solutions of silver nitrate, and is also decomposed by a warm acid solution of silver nitrate and by concentrated alkalis. It yields sodium oxalate when treated with sodium, and when acted on by phosphorus pentachloride, an amorphous chloride is obtained. It does not react with hydroxylamine or phenylhydrazine, but combines with orthotoluylenediamine to form an *azine*,

$$C_6H_3Me<\begin{matrix}N:C\cdot CCl_2\\|\\N:CHCl-\end{matrix}>C(OH)\cdot COOH,$$

which is a very unstable, crystalline compound, soluble in alcohol, ether, water, and ammonia. This azine yields another very sparingly soluble yellow azine when warmed with water. The *acetyl*-derivative is amorphous and insoluble in water, but it dissolves in soda and is at the same time reconverted into the acid.

Trichlorotriketopentamethylene, $<\begin{matrix}CO\cdot CCl_2-\\CO\cdot CHCl\end{matrix}>CO$, the third product of the action of chlorine on potassium chloranilate, is prepared

by treating a neutral solution of the preceding compound with chlorine or bromine, or by acting on potassium chloranilate with a solution of sodium hypochlorite containing free chlorine. The product is precipitated with hydrochloric acid, extracted with ether, and purified by means of the ammonium-derivative. It is a colourless, crystalline, very deliquescent substance with a strongly acid reaction, and melts at 125°. It is very stable towards hot, concentrated sulphuric acid, but is very readily oxidised to oxalic acid when treated with alkaline potassium ferricyanide or other oxidising agents. The *ammonium*-derivative, $C_5(NH_4)Cl_3O_3$, is prepared by treating the acid with alcoholic ammonia and evaporating the filtered solution at the ordinary temperature. It crystallises from alcohol in yellowish prisms, melts at 207° with decomposition, is very readily soluble, and is decomposed by silver nitrate. By concentrated alkalis, both the ammonium-derivative and the substance itself are decomposed into oxalic acid and a chloride. All the metallic derivatives are readily soluble. The *monoxime*, $C_5HCl_3O_2(NOH)$, obtained by mixing the ammonium salt with hydroxylamine hydrochloride, crystallises in striated plates melting at 123—125°. When the ammonium-derivative is treated with an excess of hydroxylamine in alkaline solution, an oily oxime is obtained. The *azine* crystallises in yellow needles. The *hydrazide*, $C_5HCl_3(N_2HPh)_3$, is crystalline.

Trichlorotribromacetone, $CBr_2Cl{\cdot}CO{\cdot}CBrCl_2$, the final product of the action of the two halogens on potassium chloranilate, is obtained when trichlorotriketopentamethylene or the hydroxy-acid is treated with an excess of bromine. It separates from ether, in which it is readily soluble, in lance-shaped crystals melting at 57°, and is insoluble in water, but slightly soluble in alcohol with decomposition. The filtrate from this product contains large quantities of oxalic acid.

Symmetrical tetrachloracetone is formed when trichlorotriketopentamethylene or the corresponding hydroxy-acid is treated with hydrochloric acid and potassium chlorate (compare Levy and Jedlicka, this vol., p. 443).

Bromanilic acid bromide, $OH{\cdot}C<\begin{matrix}CO{\cdot}CBr_2\\CBr_2{\cdot}CO\end{matrix}>C{\cdot}OH$, is prepared by adding the calculated quantity of bromine-water to a neutral, aqueous solution of potassium bromanilate, acidifying with hydrobromic acid, extracting with ether, and, after drying over anhydrous copper sulphate, evaporating the ether in a partial vacuum as quickly as possible. As a portion of the product loses bromine very readily, and is thereby converted into the original acid, the residue is treated with chloroform in which the latter is insoluble. On evaporating the chloroform, the new acid separates in colourless prisms which melt at 184—186°, and soon turn red owing to loss of bromine. The acid is very readily soluble in water, yielding a strongly acid solution which probably contains a hydrate. When treated with concentrated alkalis in aqueous solution, it is readily decomposed, yielding a product which consists principally of tribromotriketopentamethylene (see below). When treated with bromine and water, it gives perbromacetone, and when heated with concentrated sulphuric acid, it is reconverted into bromanilic acid.

Tribromotriketopentamethylene, $<^{CO\cdot CBr_2-}_{CO\cdot CHBr}>CO$, is prepared by adding a solution of potassium bromate containing free bromine to an aqueous solution of potassium bromanilate until the latter becomes colourless, destroying the excess of bromine with sodium hydrogen sulphite, and proceeding as described in the case of the corresponding chlorinated derivative. It crystallises in plates, melts at 191° with decomposition, and is more sparingly soluble in water than the corresponding chloro-ketone, which it resembles both in properties and reactions. The *ammonium* salt, $C_5(NH_4)Br_3O_3$, crystallises in needles or plates, melts at 183—184°, and is entirely converted into *hexabromacetone*, $CBr_3\cdot CO\cdot CBr_3$, melting at 110°, when treated with bromine and water.

Trichlorotriketovaleric or *trichlorodiacetylglyoxlic acid*,

$$COOH\cdot CO\cdot CHCl\cdot CO\cdot CO\cdot CHCl_2,$$

is formed by intramolecular change when trichlorodiketopentamethylenehydroxycarboxylic acid is warmed with several times its volume of concentrated sulphuric acid. It is a crystalline compound, melts at 112—114°, and is readily soluble in all ordinary solvents, but is not deliquescent. It yields an oily chloride with phosphorus pentachloride, and shows all the properties of a ketone acid forming a crystalline *oxime* and an oily *hydrazide*. The *azine*,

$$C_6H_3Me\begin{<}N{:}C\cdot CHCl_2\\ N{:}C\cdot CHCl\cdot CO\cdot COOH,\end{<}$$

crystallises in white needles and is very stable; it is readily soluble in alcohol and moderately so in ether, but almost insoluble in water. Chlorine has no action on cold, aqueous solutions of the acid, but on heating, total decomposition takes place. It is decomposed by hypochlorous acid and by a mixture of potassium chlorate and hydrochloric acid, even in very dilute solutions. When boiled with bromine and water, small quantities of trichlorotribromacetone are obtained. The *lead*, *mercurous*, and *silver* salts are sparingly soluble.

By reducing and oxidising trichlorodiketopentamethylenehydroxycarboxylic acid and trichlorodiacetylglyoxylic acid, the author has obtained a series of acids, a description of which will follow at some later date. F. S. K.

Conversion of Hydrindonaphthene- and Indonaphthene-derivatives into Substituted Acetophenonecarboxylic Acids. By T. ZINCKE and C. GERLAND (*Ber.*, **21**, 2396—2402).—*Dichloracetophenoneorthocarboxylic acid*, $COOH\cdot C_6H_4\cdot CO\cdot CHCl_2$, is obtained when dichlorodiketohydrindonaphthene is dissolved in alcohol, and concentrated potash, containing a little alcohol, is gradually added until an addition of water causes no turbidity. The solution is then acidified with hydrochloric acid, diluted with water, and the product recrystallised from hot benzene. It forms colourless, shining, monoclinic prisms, melts at 124°, is readily soluble in alcohol and glacial acetic acid, moderately soluble in benzene, and sparingly in light petroleum. It is

decomposed by soda but dissolves in sodium carbonate unchanged. The *methyl* salt, $C_{10}H_8Cl_2O_3$, forms well-defined, monoclinic crystals, and is readily soluble in alcohol and acetic acid, and moderately soluble in ether.

Trichloracetophenoneorthocarboxylic acid, already prepared by Gabriel and Michael, is obtained in small quantities when a sodium carbonate solution of chlorhydroxynaphthaquinone is treated with chlorine, but it is best prepared by passing chlorine through a sodium carbonate solution of dichlorodiketohydrindonaphthene or dichloracetophenonecarboxylic acid. It crystallises from acetic acid or dilute nitric acid in thick, shining needles melting at 142°. It is decomposed into phthalic acid and chloroform when treated with soda, but it dissolves in sodium carbonate unchanged.

Dichlorobromacetophenonecarboxylic acid, $COOH\cdot C_6H_4\cdot CO\cdot CCl_2Br$, prepared by treating the dichlorinated derivative with hypobromous acid, is very similar to the preceding compound. It melts at 150°, is not decomposed by sodium carbonate, and yields dichlorobromomethane when treated with soda.

Chlorodibromacetophenonecarboxylic acid, $COOH\cdot C_6H_4\cdot CO\cdot CClBr_2$, is formed when dibromodiketohydrindonaphthene is treated with hypochlorous acid or when the corresponding chlorobromo-derivative is treated with hypobromous acid. It melts at 153°, and is similar to the acids described above. Tribromacetophenonecarboxylic acid, already prepared by Gabriel and Michael, crystallises with 1 mol. H_2O and melts at 152—160°.

The trihalogen-acetophenonecarboxylic acids can also be obtained by treating a sodium carbonate solution of the halogen-derivative of ketohydroxyindonaphthene with halogens. The dihalogen acids are decomposed by alkaline hypobromites or hypochlorites with formation of a salt of phthalic acid and trihalogen-derivatives of methane.

F. S. K.

Action of Ethyl Isobutyrate and of other Ethereal Salts on Ethyl Oxylate. By W. Wislicenus (*Annalen*, **246**, 339—341).—Attempts to effect the combination of ethyl oxalate and ethyl isobutyrate by means of sodium or sodium ethoxide were unsuccessful.

The preparation of ethyl phenyloxalacetate has been previously described by the author (Abstr., 1887, 587). The phenylhydrazine-derivative crystallises in needles, melts at 69—70°, and dissolves freely in ether and benzene. The solution of the compound in sulphuric acid gives an intense violet-red coloration with ferric chloride.

W. C. W.

Action of Ethyl Acetate on Ethyl Phthalate. By W. Wislicenus (*Annalen*, **246**, 347—355).—The action of sodium on a mixture of ethyl acetate and phthalate has been previously described by the author (Abstr., 1887, 587). The product is the sodium-derivative of *ethyl diketohydrindonaphthenecarboxylate* (abbreviated to *diketohydrindenecarboxylate*), $C_{12}H_9O_4Na$. It is deposited from water in yellow needles containing 1 mol. H_2O, and from hot alcohol in anhydrous crystals. The aqueous solution forms yellow precipitates with barium, calcium, and several other metallic salts. The silver

salt is crystalline and the copper compound, $(C_{12}H_9O_4)_2Cu$, is deposited from a hot alcoholic solution in green crystals.

Ethyl diketohydrindenecarboxylate, $C_{12}H_{10}O_4$, is soluble in alcohol, ether, benzene, and light petroleum, and melts with decomposition at 75—78°. Ferric chloride produces a deep-red coloration in the alcoholic solution. It is decomposed by alkalis, forming alcohol, an alkaline carbonate, and *α-diketohydrindene*, $C_6H_4<\begin{matrix}CO\\CO\end{matrix}>CH_2$. The latter is best prepared by acidifying an aqueous solution of the yellow sodium compound, $C_{12}H_9O_4Na$, and extracting the mixture with ether. The residue obtained by evaporating the extract is recrystallised from light petroleum. The pure compound forms colourless needles and melts with decomposition at 129—131°. The phenylhydrazine and isonitroso-derivatives have already been described (*loc. cit.*). The dibromo-derivative, $C_6H_4.(CO)_2.CBr_2$, is identical with the product which Zincke and Gerland (this vol., p. 290) obtained by the action of bromine on bromamido-α-naphthaquinonimide.

The ethyl salt of methyldiketohydrindenecarboxylic acid is obtained by heating in sealed tubes at 120°, a mixture of methyl iodide, alcohol, and the sodium compound of ethyl diketohydrindenecarboxylate. The compound crystallises in prisms, melts at 72—74°, and dissolves freely in alcohol and ether. W. C. W.

Action of Ethyl Oxalate on Lactones. By W. Wislicenus (*Annalen*, **246**, 342—347).—The preparation of the sodium compound of ethyl hydrophthalyloxalate by the action of sodium ethoxide on an ethereal solution of ethyl oxalate and phthalide, has been previously described by the author (Abstr., 1887, 952). The sodium compound is freely soluble in water, and the solution produces precipitates in solutions of calcium, barium, lead, copper, and silver salts. Ethyl hydrophthalyloxalate has been previously described (*loc. cit.*). The phenylhydrazine-derivative crystallises in prisms and melts at 157—159°; it dissolves freely in benzene and in hot alcohol. Ethyl hydrophthalyloxalate is decomposed by an excess of hot, dilute alkali, yielding alcohol, oxalic acid, and hydroxymethylbenzoic acid. On oxidation with nitric acid, ethyl hydrophthalyloxalate splits up into oxalic and phthalic acids. W. C. W.

Action of Potassium Hypobromite on Amides. By S. Hoogewerff and W. A. van Dorp (*Rec. Trav. Chim.*, **6**, 373—388).—*Phenylsulphondibromamide*, $SO_2Ph{\cdot}NBr_2$, is best prepared by dissolving 7 grams of phenylsulphonamide in an alkaline solution of potassium hypobromite containing 21 grams of bromine and 35 grams of potash in 100 c.c. of water, and adding dilute acetic acid as long as the solution remains alkaline. The product is washed with cold water, then with water at about 75° to remove the amide present, and the residue crystallised from pure chloroform. In one experiment, 8·4 grams of the dibromamide were obtained. It separates from chloroform, in which it is readily soluble, in red crystals which melt at 115—116° or at 100° if heated for some time, and explode slightly at higher temperatures. It is unstable and is partially or entirely converted into phenylsulphon-

amide when treated with alcohol, ether, benzene, carbon bisulphide, or hot water. It is insoluble in cold water, but soluble in hot acetic acid and crystallises therefrom in small plates.

The *potassium* salt, $SO_2Ph{\cdot}NKBr$, of the bromamide is precipitated when the dibromamide is dissolved in potash, in which it is very readily soluble, and the solution mixed with concentrated potash; or when a solution of phenylsulphonamide in potassium hypobromite is treated with concentrated potash. It crystallises in small tables and decomposes with slight explosion when heated above 100°, but it is very stable in alkaline solution. It is converted into phenylsulphonamide by ammonia, and when an aqueous solution is treated with dilute acetic acid, a yellow precipitate, consisting of phenylsulphonamide and the corresponding dibromo-derivative, is obtained. The *sodium* salt, $SO_2Ph{\cdot}NNaBr$, prepared in like manner, is more sparingly soluble in water than the potassium salt and crystallises in small tables or leaves. The *silver* salt, $SO_2Ph{\cdot}NAgBr + H_2O$, crystallises in yellow prisms and is decomposed by warm water.

A compound having the composition $2(SO_2Ph{\cdot}NBr_2) + KBr$, is obtained when the potassium salt is treated with a solution of bromine in potassium bromide. It is decomposed by water.

Phenylacetobromamide, $CH_2Ph{\cdot}CO{\cdot}NHBr$, is prepared by dissolving bromine (1 mol.) in an aqueous solution of potash (4 mols. KHO), and adding phenylacetamide to the solution kept at about 20°. The solution is then neutralised with acetic acid and the product crystallised from benzene, glacial acetic acid, or pure chloroform. It can also be crystallised from pure ether, in which, however, it is only sparingly soluble. It forms colourless needles, melts and turns red at 123—125°, and soon decomposes, especially in the light, even at the ordinary temperature. Cold alcohol decomposes it only very slowly, but when heat is applied, it is partially converted into the amide and the alcohol is oxidised to aldehyde. It is decomposed by ammonia, sulphurous acid, and sodium thiosulphate, with formation of the amide. It has acid properties and is readily soluble in alkalis, but when the solution is heated it is decomposed with formation of benzylamine, potassium carbonate, and potassium bromide.

Heptylamine is best prepared by dissolving caprylamide (1 mol.) in alkaline potassium hypobromite (1 mol. bromine and 4 mols. KHO in 16 times its weight of water) and distilling as quickly as possible with superheated steam. The yield is 60 to 65 per cent. Heptylamine boils at 155—156° with the stem of the thermometer entirely in the vapour; it attracts carbonic anhydride from the air and forms with it a solid salt. The *platinochloride* decomposes between 220° and 230° and has no well-defined melting point. The *picrate*,

$$C_{17}H_{15}NH_2,C_6H_3N_3O_7,$$

crystallises from dilute alcohol in needles melting at 120—121·5°.

Octylamine is best prepared by treating pelargonamide with a solution of hypobromite similar to that described above. The compound, $NH(C_8H_{17}){\cdot}CO{\cdot}NH(C_9H_{17}O)$, (compare Hofmann, *Ber.*, **15**, 760), soon separates from the solution. Acetic acid is then added as long as a precipitate is produced, and the bromamide obtained, after

drying between blotting-paper, is mixed with lime and distilled. The yield is about 45 per cent. of the quantity which should be obtained from the bromamide employed. Octylamine boils at 179—180°, the thermometer entirely in the vapour; it attracts carbonic anhydride from the air and solidifies. The *platinochloride* decomposes above 200°, but has no definite melting point. The *picrate*, $C_8H_{17}NH_2,C_6H_3N_3O_7$, crystallises from alcohol in oblong tables and melts at 112—114°.

Phenylethylamine oxalate, $(C_8H_{11}N)_2,C_2H_2O_4$, crystallises from water in small tables and melts at 218°. The *acid* salt, $C_8H_{11}N,C_2H_2O_4$, crystallises from water in needles, melts at 181°, and is decomposed by alcohol into oxalic acid and the neutral salt (compare Abstr., 1887, 245). F. S. K.

Nitramine derived from Tetramethyldiamidobenzophenone. By P. van Romburgh (*Rec. Trav. Chim.*, **6**, 365—372; compare this vol., p. 1079).—Tetramethyldiamidobenzophenone combines with symmetrical trinitrobenzene (2 mols.), yielding a compound which crystallises in long, reddish-violet needles melting at about 100°, and with 1 mol. forming a compound which crystallises in small, deep-violet plates and melts at 123°. It also combines with metadinitrobenzene (2 mols.); the product crystallises in small plates or in compact, red crystals. These additive products are stable in the air but are at once decomposed by acids.

Tetranitrodimethyldinitramidobenzophenone,

$$NO_2\cdot NMe\cdot C_6H_2(NO_2)_2\cdot CO\cdot C_6H_2(NO_2)_2\cdot NMe\cdot NO_2,$$

is formed when tetramethyldiamidobenzophenone is gradually mixed in the cold with nitric acid of sp. gr. 1·48 and the mixture boiled; if nitric acid of sp. gr. 1·53 is employed, the tetramethyldiamidobenzophenone takes fire. The crude product turns deep-red when poured into ammonia, but it loses this property when boiled with alcohol, and the alcoholic extract contains trinitrophenylmethylnitramine melting at 127°. Tetranitrodimethylnitramidobenzophenone forms pale-yellow crystals and is insoluble in most ordinary solvents, but sparingly soluble in acetone and acetic acid. It dissolves readily in nitric acid of sp. gr. 1·53 and when treated with hot, concentrated sulphuric acid it dissolves with evolution of gas, forming a dark-brown solution. When heated in a capillary tube above 200° for some time, it begins to decompose, at about 210—215° as a rule, but the temperature of decomposition depends greatly on the manner of heating, as it is not decomposed when heated for 30 seconds at 220°. When it is boiled with dilute potash, methylamine is evolved and a dark-brown solution obtained from which sulphuric acid precipitates a yellow product soluble in boiling water and in ammonia. It is not decomposed by boiling potassium carbonate. From this last-mentioned fact (compare van Romburgh, *Rec. Trav. Chim.*, **3**, 392), and from the results obtained by Klobbie (this vol., p. 466), based on the conclusions drawn from Baither's investigations (*Ber.*, **20**, 1737, Abstr., 1887, 816), the constitution of this compound is probably $[(NMeNO_2) : (NO_2)_2 : CO = 1 : 2 : 6 : 4]$ (for both rings).

Tetranitrodimethyldiamidobenzophenone,

$$C_6H_2(NHMe)(NO_2)_2{\cdot}CO{\cdot}C_6H_2(NHMe)(NO_2)_2,$$

is obtained when the preceding compound is boiled with phenol. It separates from phenol in shining, golden crystals, melts at about 225° with decomposition, and is sparingly soluble in the ordinary solvents. It dissolves in sulphuric acid, yielding a slightly coloured solution from which it is precipitated unchanged by water. When heated with nitric acid, it is reconverted into the nitramine, and when boiled with dilute potash it yields a brown solution from which acids precipitate brown flocks soluble in ammonia. F. S. K.

Action of Nitrous Acid on Tetramethyldiamidobenzophenone. By E. Bischoff (*Ber.*, **21**, 2452—2455).—The compound $C_{17}H_{19}N_3O_2$ is obtained when tetramethyldiamidobenzophenone is diazotised in the cold, and the product precipitated with very dilute soda. It crystallises from hot alcohol in golden-yellow plates, melts at 158—159°, is readily soluble in hot alcohol, benzene, and chloroform, and soluble in ether. It has basic properties and dissolves in dilute hydrochloric acid, but is reprecipitated by water. It gives Liebermann's reaction, and is reconverted into tetramethyldiamidobenzophenone when reduced with stannous chloride and hydrochloric acid. Its constitution is probably $NMe_2{\cdot}C_6H_3{<}\begin{matrix}CO\text{---}\\N(OH)\end{matrix}{>}C_6H_3{\cdot}NMe_2$.

F. S. K.

Desoxybenzoïn and Desaurins. By E. Ney (*Ber.*, **21**, 2445—2452).—*Desoxybenzoïnhydrazide*, $CH_2Ph{\cdot}CPh{:}N{\cdot}NHPh$, crystallises in slender needles, melts at 106°, and is unstable.

When methyldesoxybenzoïn is treated with sodium ethoxide and amyl nitrite in the cold, benzoic acid and acetophenoneoxime are obtained. Desoxybenzoïn yields an orthonitro- and a paranitro-derivative when nitrated in the cold.

Paranitrodesoxybenzoïnoxime crystallises from alcohol in needles, and melts at 107°.

Paramidodesoxybenzoïnoxime crystallises from alcohol and melts at 141°.

Diazodesoxybenzoïn chloride gives brown dyes with alkaline solutions of α-naphthol and α-naphtholsulphonic acid.

Hydroxydesoxybenzoïn, $OH{\cdot}C_6H_4{\cdot}CH_2{\cdot}COPh$, prepared by diazotising the amido-derivative, crystallises from water in scales, melts at 129°, and is soluble in soda. The *sodium* salt crystallises from water in slender needles. The *acetyl*-derivative crystallises in plates and melts at 87°. When acetylhydroxybenzoïn is treated with carbon thiochloride and sodium ethoxide, a violent reaction takes place, and a yellow thiocarbonyl-derivative is obtained. It does not melt below 300°, dissolves in sulphuric acid with a carmine-red coloration, and is decomposed when boiled with alkalis. The chloroform solution has a green fluorescence.

Methoxydesoxybenzoïn, $MeO{\cdot}C_6H_4{\cdot}CH_2{\cdot}COPh$, prepared by treating the chloride of phenylacetic acid with anisoïl and aluminium chloride in carbon bisulphide solution, crystallises from alcohol, melts at 76°,

and boils at 360°. The *oxime* melts at 111°. The *benzyl*-derivative melts at 99—100°. The *methyl*-derivative, MeO·C_6H_4·CHMe·COPh, is an oil and boils at 330°. The *ethyl*-derivative crystallises in long prisms melting at 47°.

Phenylanisyldesaurin, $C_{16}H_{12}O_2S$, the thiocarbonyl-derivative of methoxydesoxybenzoïn, is prepared as described above from carbon thiochloride and the benzoïn. It is a yellow substance, dissolves in chloroform with a green fluorescence, and in concentrated sulphuric acid with a blue coloration. F. S. K.

Action of Hypochlorous and Hypobromous Acids on Chlor- and Brom-hydroxynaphthaquinone and their Conversion into Hydrindonaphthene- and Indonaphthene-derivatives. By T. Zincke and C. Gerland (*Ber.*, **21**, 2379—2395).—Dichloroketohydroxyhydrindonaphthenecarboxylic acid (this vol., p. 489) is prepared by passing chlorine through a 10 per cent. sodium carbonate solution of chlorhydroxynaphthaquinone until the solution commences to become turbid. A small quantity of hydrochloric acid is then added, the filtered solution mixed with an excess of hydrochloric acid, and the product, which separates after some time, recrystallised from hot, dilute sulphuric acid. It is also obtained when dichlorotriketohydronaphthalene is boiled with dilute hydrochloric acid. It crystallises from water in prisms melting at 127—128°, and from a mixture of light petroleum and ether in monoclinic prisms which melt generally at 131—132°, but at a higher temperature when heated quickly. The anhydrous acid, which can be obtained by heating carefully, crystallises from a mixture of ether and light petroleum, and melts first at 70°, and then at 135—136°. It yields chloroketohydroxyindonaphthene when heated with alkalis or baryta, and is decomposed by water at 180°.

Bromochloroketohydroxyhydrindonaphthenecarboxylic acid,

$$CO<\begin{matrix}C_6H_4 \text{---}\\ C(ClBr)\end{matrix}>C(OH)\cdot COOH + H_2O,$$

is prepared from bromohydroxynaphthaquinone as described above, or from chlorhydroxynaphthaquinone in a similar manner, employing bromine instead of chlorine; it is also obtained when chlorobromotriketohydronaphthalene is dissolved in sodium carbonate. It crystallises from hot hydrochloric acid in thick, colourless needles melting at 126—127° with decomposition, and from a mixture of ether and light petroleum in monoclinic prisms which melt at a slightly higher temperature. When heated, first at 100—115° and finally at 130°, the crystals lose 1 mol. H_2O, become opaque, and melt at 176—177°, but after recrystallising from a mixture of ether and light petroleum at 190°. The anhydrous acid takes up 1 mol. H_2O when crystallised from dilute hydrochloric acid. When boiled with water or treated with cold, dilute soda, it yields chloroketohydroxyhydrindonaphthene (compare Zincke, Abstr., 1887, 728). The *methyl* salt forms monoclinic crystals melting at 134—135°. The *acetyl*-derivative of the methyl salt crystallises is small, rhombic plates melting at 136—137°.

Dibromoketohydroxyhydrindonaphthenecarboxylic acid,

$$CO<\begin{matrix}C_6H_4\\CBr_2\end{matrix}>C(OH)\cdot COOH + H_2O,$$

prepared by treating a sodium carbonate solution of bromhydroxynaphthaquinone with bromine, or by dissolving dibromotriketohydronaphthalene in cold, dilute sodium carbonate, crystallises from hot hydrochloric acid in needles melting at 126—127°. It separates from a mixture of ether and light petroleum in large monoclinic crystals, which melt and give off water at 137—138°. The anhydrous acid melts at 170°, and crystallises unchanged from hot dilute hydrochloric acid, but after crystallising from a hot mixture of benzene and light petroleum, it melts at 165°, and seems to contain one of the solvents. When boiled with water, it is quickly decomposed, yielding bromoketohydroxyindonaphthene, which is also formed when the acid is dissolved in cold soda or baryta. The *methyl* salt crystallises in transparent prisms melting at 137°. The *acetyl*-derivative,

$$CO<\begin{matrix}C_6H_4\\CBr_2\end{matrix}>C(OAc)\cdot COOMe,$$

forms shining plates.

The three acids described above are very similar in appearance and behaviour, but the dichloro-compound is the most stable, and is not decomposed by cold soda.

Dichloro-α-diketohydrindonaphthene (this vol., p. 489), dissolves readily in dilute soda, and is decomposed into phthalic acid, formaldehyde, and hydrochloric acid. When treated with alcoholic potash, it is converted into dichloroacetophenonecarboxylic acid (compare this vol., p. 1193).

Chlorobromo-α-diketohydrindonaphthene (this vol., p. 290) is formed by the action of chlorine on bromoketohydroxyindonaphthene, or by the action of bromine on the corresponding chlorinated derivative. It crystallises from glacial acetic acid or alcohol in colourless plates, melts at 146—147°, and yields dibromochloromethane, chloroketohydroxyindonaphthene and phthalic acid when treated with dilute soda. Alcoholic potash decomposes it into chlorobromoform, chloroketohydroxyindonaphthene and other compounds, amongst which is probably dibromacetophenonecarboxylic acid (*loc. cit.*).

Dibromo-α-diketohydrindonaphthene (this vol., p. 290) yields bromoform, phthalic acid, and bromoketohydroxyindonaphthene when treated with alkalis. It is decomposed by alcoholic potash in a manner similar to the preceding compound.

Chloroketohydroxyindonaphthene,

$$CO<\begin{matrix}C_6H_4\\CCl\end{matrix}>C(OH) \text{ or } CO<\begin{matrix}C_6H_4\\CClH\end{matrix}>CO$$

(Zincke, *loc. cit.*), can be obtained by dissolving chlorobromoketohydroxyhydrindonaphthenecarboxylic acid in dilute alkali, and after some time precipitating the product with hydrochloric acid. It yields a trihalogen-derivative of acetophenonecarboxylic acid when treated with halogens in sodium carbonate solution, but in soda solu-

tions, under the same conditions, it is decomposed into phthalic acid and a trihalogen-derivative of methane.

Bromoketohydroxyindonaphthene, already obtained by Roser and Haselhoff, is prepared in like manner from dibromodiketohydrindonaphthene or from the corresponding dibromohydroxy-acid. It is unstable, crystallises in needles, and melts at 118°. It behaves towards alkalis and halogens like the corresponding chlorinated derivative, and in acetic acid solution is easily converted into the dihalogen diketone. F. S. K.

α-Naphthylamine-δ-monosulphonic Acid. By R. Hirsch (*Ber.*, **21**, 2370—2373).—*α-Naphthylamine-δ-monosulphonic acid* is obtained when α-naphthylamine is heated at 125—130° for eight to nine hours with 5 parts of concentrated, but not fuming, sulphuric acid. The acid formed in the reaction is converted into the calcium salt, the latter decomposed with hydrochloric acid, and the pure compound obtained in rhombic plates by extracting repeatedly with small quantities of water and recrystallising several times from boiling water. When hydrochloric acid is added to a 1 per cent. boiling solution of the sodium salt, the acid separates in long needles, which gradually dissolve as the solution becomes cold, and change more or less completely into the rhombic plates. The acid dissolves in about 150 parts of boiling, or 450 parts of cold water, but is more sparingly soluble in alcohol and insoluble in ether and benzene. The *sodium* and *ammonium* salts are very readily soluble in water; the sodium salt separates when a solution of either salt is saturated with sodium chloride. The *potassium* and *magnesium* salts are crystalline and sparingly soluble in cold water. The diazo-derivative yields a dinitronaphtholsulphonic acid when boiled with dilute nitric acid; the *potassium* salt of this sulphonic acid is readily soluble in water, but the *barium* salt is only sparingly soluble. F. S. K.

An Isomeride of Oximidonaphthol. By S. Meerson (*Ber.*, **21**, 2516—2518; compare this vol., p. 713).—Acetamidonaphthaquinone is heated with 20 parts of strong sulphuric acid on a water-bath for one hour, the *amidonaphthaquinone* precipitated with water and crystallised from alcohol, from which it separates in lustrous, brown plates, melting at 200°. The *sulphate* forms reddish needles. When oximidonaphthol (Graebe and Ludwig, *Annalen*, **154**, 318) and amidonaphthaquinone are heated with acetic anhydride, they both yield the same *triacetylamidonaphthaquinol*, $C_{10}H_5(OAc)_2{\cdot}NHAc$. This, by treatment with cold sulphuric acid, is converted into acetamidonaphthaquinone. By means of this reaction it is therefore possible to convert oximidonaphthol into its isomeride. N. H. M.

Intramolecular Change of Phenanthraquinonemonoxime. By P. Wegerhoff (*Ber.*, **21**, 2355—2357).—*Diphenimide*,

$$C_6H_4<\begin{matrix}CO{\cdot}NH{\cdot}CO\\ -C_6H_4-\end{matrix}>,$$

is formed when phenanthraquinonemonoxime is treated with hydro-

chloric acid according to Beckmann's method (this vol., p. 43). It crystallises in colourless needles, melts at 215°, and is readily soluble in water, but only sparingly in alcohol.

Diphenamic acid, obtained by treating the imide with water, melts at 187—188°, and is soluble in hot water.

Diphenamide, prepared by acting on the imide with ammonia, loses ammonia when heated, and melts at the same temperature as the imide; it is soluble in water.

Diphenyleneketonecarboxylamide, $C_6H_4<\overset{CO}{\underset{__}{}}>C_6H_3\cdot CONH_2$, is obtained when phenanthraquinonemonoxime is heated with concentrated sulphuric acid. It crystallises from absolute alcohol with $\frac{1}{2}$ mol. alcohol, in slender, silky needles, melting at 225°. This compound is also formed when the chloride of diphenyleneketonecarboxylic acid is treated with anhydrous ammonia, or when diphenimide is warmed with concentrated sulphuric acid. Phenanthraquinonemonoxime also undergoes intramolecular change when treated with phosphorus pentachloride, acetic chloride, or acetic anhydride.

F. S. K.

Anthracene Hydride. By L. Lucas (*Ber.*, **21**, 2510—2511).—*Perhydroanthracene*, $C_{14}H_{24}$, is obtained by heating pure anthracene (1·5 gram) with an equal weight of amorphous phosphorus and hydriodic acid, sp. gr. = 1·7 (8 grams), for 12 hours at 250°. It crystallises in colourless plates, melts at 88°, boils at about 270°, and is readily soluble, except in water; it distils readily with steam. Bromine dissolved in carbon bisulphide scarcely acts on it.

When, in the reduction of anthracene, the heating is only continued for 5 to 6 hours, two hydrocarbons, probably of the formula $C_{14}H_{20}$, are obtained. The one is crystalline and melts at 52°, the other is an oil.

N. H. M.

Derivatives of Dianthranyl. By H. Sachse (*Ber.*, **21**, 2512—2514).—*Dianthranyl tetrahydride*, $CH_2<\overset{C_6H_4}{\underset{C_6H_4}{}}>CH\cdot CH<\overset{C_6H_4}{\underset{C_6H_4}{}}>CH_2$, is obtained by boiling dianthranyl (2 grams) with 4 per cent. sodium amalgam (150 grams) for some hours. It crystallises from benzene in prismatic needles melting at 248—249°, and dissolves rather sparingly in alcohol, readily in hot benzene. Bromine acts on it with formation of dibromanthracene. When dianthranyl is heated with hydriodic acid and phosphorus at 200°, anthracene dihydride (m. p. 106°) is formed.

Dibromodianthranyl, $C_{28}H_{16}Br_2$, is formed when bromine, diluted with glacial acetic acid, is slowly added to a boiling solution of dinitrodianthranyl in glacial acetic acid. It crystallises in lemon-coloured plates, rather easily soluble in benzene. It melts much above 300°.

Dichlorodianthranyl, $C_{28}H_{16}Cl_2$, obtained by heating dinitrodianthranyl with hydrochloric acid (sp. gr. = 1·19) at 180°, crystallises from glacial acetic acid in matted needles of a golden lustre which do not melt below 300°. It is rather readily soluble in benzene, sparingly in glacial acetic acid, very sparingly in alcohol. The solutions show a

blue fluorescence. Both the bromo- and the chloro-derivatives are sensitive to light. N. H. M.

Derivatives of Anthranol. By F. GOLDMANN (*Ber.*, **21**, 2505—2508).—*Ethoxyethylanthranyl*, $C_6H_4\langle\begin{matrix}C(OEt)\\|\\CEt—\end{matrix}\rangle C_6H_4$, is formed as a third product in the reaction between ethyl iodide, potash, and anthranol (this vol., p. 714), and is separated by means of the picrate. It crystallises from dilute alcohol in pale yellow, slender needles, melts at 77°, and dissolves very readily in benzene, light petroleum, and ether; the solutions show a blue fluorescence. The *picrate* forms reddish-brown needles. When ethoxyethylanthranyl is oxidised with chromic acid, Liebermann's ethyloxyanthranol is formed. The presence of anthranol can be shown by adding diazobenzenesulphonic acid to the dilute alkaline solution, when the solution acquires an intense blue colour. The dye may be recovered by converting it into the lead salt, which is then decomposed by hydrogen sulphide; it is obtained as a red, amorphous mass, readily soluble in water, with orange colour. N. H. M.

Derivatives of Anthranol. By F. HALLGARTEN (*Ber.*, **21**, 2508—2510).—*Dimethylanthrone*, $C_6H_4\langle\begin{matrix}CO—\\CMe_2\end{matrix}\rangle C_6H_4$, is prepared by boiling anthranol (12 grams) with potash (12 grams), water (60 c.c.), and methyl iodide (40 grams) for $3\frac{1}{2}$ hours in a reflux apparatus. The red oil is washed, boiled with water, and extracted with ether, from which it is precipitated by light petroleum. It crystallises in pale yellow, clear crystals, melts at 93—94°, dissolves readily in benzene and ether, sparingly in light petroleum.

Dimethylanthracene hydride, $C_6H_4\langle\begin{matrix}CH_2—\\CMe_2\end{matrix}\rangle C_6H_4$, obtained by heating the dimethylanthrone with hydriodic acid (sp. gr. 1·7) and amorphous phosphorus at 140—150°, is a white, crystalline substance which melts at 56° and dissolves in ether, benzene, and glacial acetic acid.

Dibenzylanthrone, $C_6H_4\langle\begin{matrix}CO———\\C(C_7H_7)_2\end{matrix}\rangle C_6H_4$, is formed when anthranol (1 part) is heated with aqueous potash (1 part of potash in 5 parts of water) and benzyl chloride (2 parts). It crystallises from benzene in lustrous, white crystals, melting at 217°; it is insoluble in light petroleum and alkalis, sparingly soluble in benzene. When reduced with hydriodic acid and amorphous phosphorus at 190°, the *hydride*, $C_6H_4\langle\begin{matrix}CH_2———\\C(C_7H_7)_2\end{matrix}\rangle C_6H_4$, is obtained. This forms slender, white crystals, readily soluble in ether, less so in benzene and alcohol; it melts at 115°.

Amylhydroanthrone, $C_6H_4\langle\begin{matrix}CO———\\CH(C_5H_{11})\end{matrix}\rangle C_6H_3$, is obtained by boiling anthranol (10 grams), potash (10 grams), water (50 c.c.), and amyl iodide (44 grams), for eight hours in a reflux apparatus. It forms

yellowish crystals, melts at 252—253°, dissolves readily in ether, sparingly in alcohol and light petroleum. The dilute solutions show a blue fluorescence. N. H. M.

Spectra of the Alkoxy-anthraquinones. By C. LIEBERMANN (*Ber.*, **21**, 2527).—The following numbers show the wave-lengths in millionths of a mm., and were obtained with solutions in cold concentrated sulphuric acid:—

Alizarin	605	493	
Alizarin ethyl ether..	598	487	
Anthraflavic acid.....	495	463	
Dimethyl salt.....	501	473	(437 very feeble)
Diethyl salt	504	477	(439 „)
Quinizarin.....	551	509	(483 feeble)
Monethyl salt	564	520	(484 „)
Diethyl salt	577	535	(494 „)
Isoanthraflavic acid....	—	540	(494 not sharp)
Diethyl salt	—	505	(492 „)
Flavopurpurin	533	495	
Diethyl salt	542	501	
Anthragallol.....	525	492	
Monethyl salt	—	515	(not sharp)
Diethyl salt.....	—	515	(„)
Rufigallic acid	576	532	
Triethyl salt.....	579	545	

(Compare Liebermann and Jellinek, this vol., p. 715.)

N. H. M.

A New Dihydrazanthraquinone: Hystazarin. By C. LIEBERMANN (*Ber.*, **21**, 2501—2502).—See next Abstract.

Hystazarin. By A. SCHOELLER (*Ber.*, **21**, 2503—2505).—*Hystazarin* (dihydroxyanthraquinone), $[(OH)_2 = 2 : 3]$, is prepared by heating catechol (5 grams), phthalic anhydride (6·5 grams), and strong sulphuric acid (75 grams), at 140—150° for 4½ to 5 hours; the product is poured into water (750 c.c.), heated to boiling and filtered hot. The precipitate is extracted with boiling water, dissolved in dilute potash and precipitated with acid. It is then dried on tiles, extracted with alcohol, and the solution evaporated. The orange-yellow mass which separates is dried and extracted with boiling benzene, which removes the small amount of alizarin formed in the reaction. The undissolved portion is recrystallised from acetone. Hystazarin forms orange-yellow, slender needles, soluble in hot alcohol, ether, and glacial acetic acid, almost insoluble in benzene; it does not melt at 260°. Alkalis dissolve it with a cornflower blue, ammonia with a violet, and strong sulphuric acid with blood-red coloration. The *barium* and *calcium salts* are blue and violet respectively. It gives a green coloration with ferric chloride. It dyes mordants only slightly. The absorption-spectrum of the sulphuric acid solution shows no bands but only a one-sided absorption. The dilute soda solution is blue, and shows an absorption of red, yellow,

and violet; on diluting, a band appears in the yellow part of the spectrum, which separates on further dilution into two bands: $\lambda = 619{\cdot}8$, $\lambda_1 = 587{\cdot}4$. The *diacetyl-derivative*, $C_{14}H_6O_2(OAc)_2$, crystallises in yellow needles melting at 205—207°. When hystazarin is reduced with zinc-dust, it is converted into anthracene. N. H. M.

Purification of Flavopurpurin. By G. Jellinek (*Ber.*, **21**, 2524—2526).—500 grams of the powdered technical product is suspended in about 3 litres of water, made alkaline with potash and filtered. The solution is diluted with water to 15 litres, heated to boiling in an open vessel, and a solution of lead acetate added until a sample of the filtered liquid gives a precipitate on the further addition of lead acetate, which becomes of a light leather colour, after carbonic anhydride is passed through. The precipitated lead compound is filtered through a bag, the bag tied up and boiled repeatedly with water in a copper vessel until the water has only a light rose colour. The contents of the bag are now decomposed by sulphuric acid and separated from lead sulphate by means of moderately dilute alcohol. The alcoholic solution is evaporated down, the first separation removed, and the rest left to crystallise.

In purifying small amounts of the technical product (50 grams and less), an alcoholic-alkaline solution is used for the precipitation of the lead compound.

The red filtrate from the lead compound yields splendid ruby-coloured needles, which, when treated with acid become converted into a mass of yellow needles of dihydroxyanthraquinone.

N. H. M.

Terpenes and Ethereal Oils. By O. Wallach (*Annalen*, **246**, 221—239).—The oil obtained from the needles of *Pinus picea* and of other species of pines consists chiefly of pinene (lævogyrate), and also contains a considerable quantity of *limonene*, which is a physical isomeride of the limonene contained in cummel oil and in the oil from orange-peel. This variety of limonene boils at 175—176°. Its sp. gr. at 20° is 0·846. It is lævogyrate, $[\alpha]_D = -105°$. The index of refraction for the sodium line is 1·47459 at 20°.

The *tetrabromide*, prepared by acting on a mixture of the limonene with glacial acetic acid, melts at 104°, and resembles the ordinary tetrabromide in crystalline form, but differs from it in its action on polarised light, as it is lævogyrate. The nitrosochloride from the lævogyrate limonene only differs from the ordinary limonene nitrosochloride by being lævogyrate.

Dipentene is formed by mixing together lævo- and dextro-limonene in equal quantities. By brominating this mixture, dipentene tetrabromide is obtained. This compound can also be prepared by mixing in equal quantities dextro- and lævo-limonene tetrabromides. Lævogyrate limonene nitrosochloride, on decomposition with alcoholic potash, yields dextrogyrate carvoxime melting at 72°. Isonitrosodipentene, which melts at 93° and is optically inactive, is produced when equal quantities of + and − carvoxime are mixed.

Isonitrosodipentene and dextrocarvoxime have identical molecular weights, according to the determinations made by Raoult's method.

The phellandrene from *Phellandrium aquaticum* and many other plants is dextrogyrate, but that from *Eucalyptus amygdalina* is lævogyrate. The nitrites from the two isomerides melt at 103°, and appear to be identical in their properties. The dextrophellandrene yields a lævogyrate nitrite, and the lævophellandrene yields a dextrogyrate nitrite. An optically inactive modification is obtained by mixing the nitrites in equal quantities. W. C. W.

Terpenes and Ethereal Oils. By O. Wallach and E. Gildmeister (*Annalen*, **246**, 265—284).—*Cineolic acid*, $C_{10}H_{16}O_5$, is prepared by oxidising cineol with potassium permanganate. The filtered solution is evaporated to dryness and the residue extracted with alcohol, which dissolves out potassium cineolate, from which the acid is easily isolated. Cineolic acid is soluble in 70 parts of water at 15°, and in 15 at 100°. It is more soluble in warm alcohol and ether. The calcium salt, $CaC_{10}H_{14}O_5 + 4H_2O$, is insoluble in boiling water, but dissolves slowly in cold water. The ethyl salt boils at 155° under 11 mm. pressure. Cineolic acid melts at 196° with decomposition. On oxidation with potassium permanganate, it yields oxalic and carbonic acids.

The oil of *Eucalyptus amygdalina* contains cineol and phellandrene. It is lævogyrate, boils between 165—180°, and its sp. gr. is 0·855 at 18°. This phellandrene is a physical isomeride of the phellandrene contained in *Phellandrium aquaticum* (see preceding Abstract). The hydrocarbon eucalyptene from *Eucalyptus globulus* is identical with dextropinene. W. C. W.

Chemistry of Buchu Leaves. By Y. Shimoyama (*Arch. Pharm.* [3], **26**, 403—417).—Flückiger obtained from the oil of buchu leaves a peculiar compound, described under the name of diosphenol; it forms colourless crystals belonging to the monoclinic system, which can be obtained several centimetres long by sublimation. It is easily soluble in alcohol, less so in ether, and scarcely at all in water. Its solutions are neutral. Diosphenol dissolves in concentrated sulphuric acid; on saturating the solution with barium carbonate and evaporating the filtrate, a little amorphous barium salt is obtained. According to Spica, diosphenol is an oxycamphor of the composition $C_{10}H_{16}O_2$; the author's results confirm this. *Methyldiosphenol*, obtained by the action of potassium hydroxide and methyl iodide on diosphenol, is a colourless liquid which boils at 232—235°, and has a sp. gr. of 0·985 at 15°. It is easily soluble in alcohol and ether, but not in water. Its composition is $C_{10}H_{13}O_2Me$. The corresponding *ethyl*-compound is also a colourless liquid insoluble in water, easily soluble in alcohol and ether, of sp. gr. 0·967 at 15°. Boiling point 270—272°. *Acetyldiosphenol* is obtained by mixing diosphenol with anhydrous sodium acetate and excess of acetic anhydride, and heating at 145° in a closed tube. The rectified product is a colourless, odourless liquid which boils at 269—270°, although not without decomposition. Sp. gr. 1·032 at 20°; easily soluble in alcohol and ether but not in water. The compound is neutral, but after rectification has an acid reaction.

Long digestion of diosphenol with alcoholic potash partly converts it into *diolic acid;* this is separated from the liquid residue in the retort by the addition of hydrochloric acid, dissolved in ammonium carbonate, treated with animal charcoal, and precipitated with acid. Diolic acid forms white, crystalline needles; its aqueous solution has an acid reaction; it neutralises strong bases and liberates carbonic anhydride from carbonates. It melts at 96—97°, volatilises slowly on the water-bath, and decomposes when heated strongly, evolving a menthol-like odour. It is soluble in 122·7 parts of water at 18°, and in 82 parts at 100°; easily soluble in alcohol, ether, chloroform, benzene, and carbon bisulphide. Its composition is $C_{10}H_{18}O_3 + H_2O$. The barium salt, $(C_{10}H_{17}O_3)_2Ba + 5H_2O$, is soluble in 67·89 parts water at 17·5° and 19·7 parts at 100°, and also in alcohol. Over sulphuric acid, it gradually loses its water of crystallisation. The white, amorphous silver salt, $C_{10}H_{17}O_3Ag$, rapidly blackens on exposure to the light. It is scarcely soluble in water even at 100°. The sodium and ammonium salts are amorphous. The calcium and magnesium salts are white, amorphous powders insoluble in water, whilst the strontium salt is easily soluble. The copper salt is light-brown, the iron salt red-brown; both are only slightly soluble in water. Diosphenol when fused with potassium hydroxide yielded an acid agreeing in every particular with diolic acid, but melting at 86°, that is 10° lower; attempts to raise the melting point of the new acid by recrystallisation were unsuccessful.

Reduction of diosphenol in alcoholic solution by means of sodium amalgam gave an oily substance, which was dissolved in aqueous ether and treated with sodium to reduce the diosphenol still remaining unacted on. The solution was then allowed to evaporate, when an oily liquid permeated with crystals was left. The crystals, prismatic in form, melt at 159°, are odourless, sparingly soluble in alcohol and ether, and the alcoholic solution does not give the dirty-green coloration with ferric chloride solution which diosphenol does. Its composition is $C_{10}H_{18}O_3$, and must be considered as the *diol alcohol.* The oily compound occurring with these crystals is probably $C_{10}H_{18}O$, the principal component of buchu oil, according to Flückiger, and described by Spica under the name of diosmeleoptene. Diosphenol dissolved in carbon bisulphide and treated with bromine gives fine yellow crystals of the composition $C_{10}H_{14}Br_2O_2$; these melt at 43°, and are soluble in alcohol and ether, but not in water. J. T.

Apiole. By J. Ginsberg (*Ber.*, **21**, 2514—2516; compare this vol., p. 722).—*Tribromapiole*, $C_{12}H_{11}Br_3O_4$, is prepared by slowly adding bromine (2 parts) dissolved in carbon bisulphide to a cooled solution of apiole (1 part) in carbon bisulphide. The crystalline mass obtained by evaporating the carbon bisulphide is washed with dilute alcohol and recrystallised from absolute alcohol, from which it separates in flat needles melting at 88—89°. It is readily soluble in most solvents except water. The solution in strong sulphuric acid is violet.

Tribromisapiole, $C_{12}H_{11}Br_3O_4$, is prepared in a manner similar to the compound just described, except that glacial acetic acid is used as the

solvent. It crystallises from glacial acetic acid in colourless, lustrous plates, melts at 120°, and resembles its isomeride in solubility.

A *chlorine-derivative* of the formula $C_{12}H_{12}O_4Cl_2$, or $C_{12}H_{14}O_4Cl_2$, was obtained by boiling a solution of isapiole in benzene with phosphorus pentachloride: it crystallises in small prisms. No crystalline chlorinated compound could be obtained from apiole.

When the nitro-derivative of apiole, previously described, is dissolved in 20 parts of bromine, the compound $C_{10}H_8Br_4O_2$ is formed. This crystallises from absolute alcohol in thin plates of a silvery lustre, melts at 159°, and is soluble in the usual solvents, except water. Isapiole, when oxidised with manganese peroxide and sulphuric acid, yields an acid melting at 250°, not identical with that obtained by Ciamician and Silber. N. H. M.

Methysticin. By D. Davidoff (*J. Russ. Chem. Soc.*, 1887, 522—523).—The author has studied the properties of methysticin from the root of Kawa-Kawa, or *Macropiper methysticum*, and some of its derivatives. It melts at 138—139°, and has the formula $C_{16}H_{18}O_5$. The diacetyl-derivative, $C_{20}H_{22}O_7$, melts at 122—123°, the dibenzoyl-compound, $C_{30}H_{26}O_7$, melts at 148°, the hydrate $C_8H_{10}O_3$ melts at 158—159°, the ethyl ether, $C_8H_9EtO_3$, melts at 99—100°, benzoylmethysticic acid, $C_{15}H_{14}O_4$, melts at 122°, and the amido-compound $C_8H_{11}NO_2$, forms yellow crystals. B. B.

α-γ-Dimethylparatoluquinoline. By W. Pfitzinger (*J. pr. Chim.* [2], **38**, 40—48).—*α-γ-Dimethylparatoluquinoline*,

$$C_6H_3Me\begin{<}CMe{:}CH\\ -N{:}CMe-\end{>},$$

is prepared as follows:—A mixture of 30 grams of paraldehyde with 40 grams of acetone is saturated in the cold with hydrogen chloride, kept for about 12 hours in a freezing mixture, then poured into a mixture of 65 grams of paratoluidine with 130 grams of fuming hydrochloric acid and heated for 6 to 8 hours, the whole being frequently shaken; the product is diluted with water, boiled to expel neutral compounds, saturated with alkali and distilled with steam. The mixture of bases in the distillate is dissolved in alcohol, precipitated with an alcoholic solution of picric acid, the picric compound decomposed with soda, and the free base distilled with steam.

It is sparingly soluble in water, and separates from the hot aqueous solution in the form of an oil which solidifies to slender, colourless needles melting at 63—64°. The crystals contain water which they lose in a desiccator but absorb again on exposure to the air. It is readily soluble in alcohol, ether, benzene, acetone, chloroform and light petroleum, and crystallises from 50 per cent. alcohol in needles which probably contain alcohol. The anhydrous base, which is somewhat difficult to obtain, boils at 277—278°, and on cooling, the greater portion solidifies to a mass of deliquescent plates which after exposure to the air melt at 63—64°. It has a sweetish smell, a sharp, bitter taste and is not acted on by nitrous acid.

The *sulphate*, $C_{12}H_{13}N,H_2SO_4 + H_2O$, crystallises from boiling

alcohol in colourless needles or large, rhombic forms which lose their water at 100—110°. The anhydrous salt is readily soluble in water but only sparingly in cold alcohol, and melts at 221—222° when heated quickly. The *hydrochloride*, $C_{12}H_{13}N,HCl + 2H_2O$, crystallises in colourless needles, loses its water at 100—110°, and is very readily soluble in alcohol and hot water. It does not melt when heated to 240°. The *platinochloride*, $2C_{12}H_{13}N,H_2PtCl_6 + 2H_2O$, forms bright-yellow needles, and is very sparingly soluble in water, but almost insoluble in alcohol and ether. It loses its water at 125—130°; the anhydrous salt is hygroscopic. The *dichromate*, $2C_{12}H_{13}N,H_2Cr_2O_7$, crystallises from hot water in long, red needles, is sparingly soluble in cold water, and decomposes gradually above 150°. The *picrate*, $C_{12}H_{13}N,C_6H_3N_3O_7$, crystallises from hot alcohol or hot acetone in greenish-yellow needles which melt at 200—201° if heated quickly.

The *methiodide*, $C_{12}H_{13}N_4MeI + H_2O$, prepared by dissolving the base in methyl iodide, crystallises from hot water in slightly coloured, shining needles, which lose their water at 100—110° and melt at 225—226° when heated quickly. It is readily soluble in alcohol and water, but insoluble in ether. Compare Combes (*Compt. rend.*, **106**, 142—145; this vol., p. 504). F. S. K.

A β-Lactone of the Quinoline Series. By A. Einhorn and P. Lehnkering (*Annalen*, **246**, 160—179).—The best yield of quinolylacrylic acid is obtained by the action of alcoholic potash or soda on the condensation product of chloral and quinaldine, trichlor-α-hydroxypropylquinoline, $C_9NH_6\cdot CH_2\cdot CH(OH)\cdot CCl_3$. 250 grams of a 20 per cent. solution of potassium hydroxide in alcohol and 50 grams of the condensation product are brought into a flask of 5 litres capacity. The mixture is placed on the water-bath. As soon as a lively reaction begins, the flask is removed and the contents are agitated. The heat produced by the reaction causes the greater part of the alcohol to evaporate and potassium quinolylacrylate remains in solution. The free acid is prepared from the potassium salt in the usual way by decomposition with hydrochloric acid.

Trichloropropenylquinoline, $C_9NH_6\cdot CH{:}CH_2\cdot CCl_3$, is prepared by gradually adding phosphorus pentachloride to trichlor-α-hydroxypropylquinoline and chloroform and heating the mixture. The chloroform is removed by evaporation and the residue is poured into water. On recrystallisation from ethyl acetate, the trichloropropenylquinoline forms prismatic plates which melt at 145°.

Quinolylacrylic acid dissolves in acetic acid saturated with hydrogen bromide. The operation is carried on in sealed tubes at 100°. On cooling, the solution deposits crystals of the *hydrobromide* of *quinolyl-β-bromopropionic acid*, $C_9NH_6\cdot CHBr\cdot CH_2\cdot COOH,HBr$. This compound is decomposed by alkaline carbonates, yielding quinolyl-β-lactic acid and the corresponding lactone if the solution is cold, or quinolylethylene with a hot solution. The *lactone* of quinolyl-β-hydroxypropionic acid, $C_9NH_6\cdot CH<\begin{matrix}CH_2\\-O-\end{matrix}>CO$, crystallises in needles, melts at 83° and decomposes at 100°. It is soluble in abso-

lute alcohol, benzene and ether, and forms crystalline salts with picric, hydrochloric, and sulphuric acids. It is converted into the quinolyl-β-lactic acid by the action of caustic alkalis, and into the corresponding amide by the action of alcoholic ammonia.

Quinolylethylene, $C_9NH_6{\cdot}CH{:}CH_2$, is an oily liquid volatile in a current of steam and slightly soluble in water. The *platinochloride*, $(C_{11}H_9N)_2,H_2PtCl_6 + 4H_2O$, forms orange-coloured needles and melts at 186—187°. The *aurochloride*, $C_{11}H_9N,HAuCl_4$, crystallises in yellowish-red needles. It melts at 158—159°.

A solution of the hydrobromide of quinolyl-β-bromopropionic acid, in excess of ammonia, deposits *quinolyllactamide*,

$$C_9NH_6{\cdot}CH(OH){\cdot}CH_2{\cdot}CONH_2,$$

in white crystals. The base melts at 151—152°. By boiling with hydrochloric acid, it is converted into the hydrochloride of *quinolyl-β-lactic* acid, $C_9NH_6{\cdot}CH(OH){\cdot}CH_2{\cdot}COOH$. This salt crystallises in thick prisms and melts at 187—188°. The *platinochloride*,

$$(C_{12}H_{11}O_3N)_2,H_2PtCl_6,$$

forms yellowish-red prisms. It melts at 218° with decomposition. Quinolyllactic acid is obtained in large, colourless prisms by covering the acid with hot chloroform, then adding methyl alcohol until the acid dissolves, and evaporating the solution. The acid melts at 176° and is soluble in methyl alcohol, ethyl alcohol, and ethyl acetate. The sodium salt exists as a jelly and also as a crystalline powder. The *methyl* salt melts at 62°, crystallises in transparent prisms, and dissolves in benzene, alcohol, and ethyl acetate. W. C. W.

Opianic Acid. By A. Bistrzycki (*Ber.*, **21**, 2518—2524).—*Diphenylhydrazonopianic acid*, $C_{22}H_{20}N_2O_4$, is obtained by adding an aqueous solution of diphenylhydrazine hydrochloride (1 mol.) containing a trace of hydrochloric acid to a concentrated, boiling aqueous solution of opianic acid (1 mol.) and sodium acetate (1 mol.), and boiling the mixture for a few minutes. It forms yellow crystals, melts at 171—172°, dissolves readily in hot alcohol, chloroform, acetone and benzene, but is almost insoluble in light petroleum. Dilute alkali solutions dissolve it readily. The *calcium salt* with 8 mols. H_2O crystallises in plates.

Diphenylhydrazonenitropianic acid, $C_{22}H_{19}N_3O_6$, is prepared in a manner similar to the opianic acid derivative, except that twice the amount of sodium acetate is used; it crystallises in small, lustrous, orange-coloured prisms, melts at 217°, dissolves readily in hot acetone, chloroform, glacial acetic acid and alcohol, sparingly in benzene and ether, and is insoluble in light petroleum. The *calcium salt* forms red, microscopic needles.

Opianylhydrazobenzene, $C_{22}H_{20}N_2O_4$, is formed when hot, concentrated alcoholic solutions of opianic acid and hydrazobenzene are mixed and boiled for a few minutes. It crystallises in white plates, melting at 186—188°, and is rather soluble in hot alcohol, acetone, and chloroform, almost insoluble in ether and light petroleum.

Benzidylopianic acid, $C_{32}H_{28}N_2O_6$, obtained from opianic acid

(2 mols.) and benzidine (1 mol.), crystallises in microscopic needles, is almost insoluble in the usual solvents, and does not melt at 230°; it can be crystallised from boiling nitrobenzene. Hot soda solution dissolves it readily.

Opiancarbamide, $C_{11}H_{12}O_5N_2$, crystallises in groups of needles, melts at 259° with decomposition, dissolves sparingly in hot alcohol, and is almost insoluble in other solvents. Hot aqueous sodium carbonate and cold potash dissolve it readily.

Toluyleneopiamine, prepared from opianic acid, metaparabenzylenediamine hydrochloride and sodium acetate, crystallises in small needles, melts at 242—243°, dissolves rather readily in boiling glacial acetic acid, acetone, and chloroform, but is almost insoluble in water, cold alcohol, and benzene. Acids and alkalis dissolve it readily.

N. H. M.

Isatropylcocaïne. By C. Liebermann (*Ber.*, **21**, 2342—2355; compare Hesse, Abstr., 1887, 1126).—An alkaloïd which the author names isatropylcocaïne is present in the mixture of amorphous alkaloïds obtained as a bye-product in the preparation of cocaïne.

Isatropylcocaïne, $C_{19}H_{23}NO_4$, is prepared in the pure state as follows: the yellow, sticky, amorphous mixture is dissolved in hydrochloric acid and the filtered solution extracted with ether. The extract contains considerable quantities of benzaldehyde. The solution is freed from ether by means of a stream of air and the base precipitated fractionally with alkali. It is then obtained in the form of a white, chalky, amorphous powder, the quantity of which is about 70 per cent. of the crude material. The middle fractions are dissolved in hydrochloric acid, again fractionally precipitated with soda, extracted with light petroleum to remove traces of cocaïne, and dried in a stream of air for several days at the ordinary temperature, and finally at 45°.

The alkaline filtrate from the alkaloïd contains ecgonine, the quantity of which is from 1 to 2 per cent. of the crude material.

Isatropylcocaïne is very similar to cocaïne, but both the base and its salts are amorphous, and the former is not so readily soluble in ammonia and light petroleum as cocaïne. It is easily soluble in cold alcohol, ether, benzene, and chloroform, but only very sparingly in light petroleum, and in all cases a resinous mass is obtained when the solvent is evaporated. It begins to soften at 65°, but has no definite melting point; at 90—100° it loses water slowly, and at 120° it turns brown and is gradually decomposed. All the salts are amorphous and soluble in water. Picric acid produces a yellow, chromic acid an orange, and potassium permanganate a violet precipitate, which changes to brown. The chlorides of mercury, tin, gold, and platinum and most of the usual reagents for alkaloïds also give amorphous precipitates. The alcoholic solution does not show an alkaline reaction with phenolphthaleïn, and is dextrorotatory.

The base is a powerful poison, but its action is not similar either to that of cocaïne or atropine; the symptoms of poisoning sometimes observed after the administration of impure cocaïne may be due to this alkaloïd.

Methyl chloride is evolved when the base is heated with con-

centrated hydrochloric acid, and when boiled with strong hydriodic acid methyl iodide is obtained, the quantity of which, estimated by Zeisel's method, showed that one methyl-group has been eliminated. It is also decomposed, with separation of methyl alcohol, when heated for a long time with dilute sulphuric acid, but no ethyl alcohol is formed. When boiled for about an hour with hydrochloric acid, sp. gr. 1·1, it is decomposed into methyl alcohol, ecgonine, and isatropic acid in molecular proportion as in the following equation:

$$C_{19}H_{23}NO_4 + 2H_2O = CH_4O + C_9H_{15}NO_3 + C_9H_8O_2.$$

The product of the reaction is filtered and the two isatropic acids in the residue separated by means of their barium salts.

γ-Isatropic acid, $C_9H_8O_2$, forms about two-thirds of the residue. It crystallises from 50 per cent. alcohol in small, colourless needles, melts at 274°, and is soluble in hot glacial acetic acid, but only very sparingly in ether, benzene, and carbon bisulphide. The *barium* salt, $(C_9H_7O_2)_2Ba$, is crystalline, and readily soluble in water. The *calcium*, *copper*, and *silver* salts are soluble in water. The ethyl- and methyl-derivatives are very readily formed when hydrogen chloride is passed into a solution of the acid in the corresponding alcohol. The *ethyl* salt, $C_9H_7EtO_2$, is insoluble in ammonia and sparingly soluble in alcohol, from which it crystallises in needles melting at 146°. *Methyl γ-isatropate*, $C_9H_7MeO_2$, crystallises in plates or needles, melts at 174°, and distils at about 300° with very slight decomposition, but has no constant boiling point. A vapour-density determination in anthracene vapour showed that it is at first polymeric, and is only completely transformed into the simple molecule after about half an hour's heating.

δ-Isatropic acid, $C_9H_8O_2$, forms about one-third of the residue. It is more easily soluble in water than the γ-acid, and melts at 206°. Neither this nor the γ-acid yields benzaldehyde when oxidised with potassium permanganate or chromic acid. An aqueous solution of the ammonium salt gives precipitates with calcium chloride, mercuric chloride, and copper acetate. The *barium* salt, $(C_9H_7O_2)_2Ba$, is sparingly soluble in water. The *silver* salt, $C_9H_7O_2Ag$, is amorphous but becomes crystalline when boiled with water. The *ethyl* salt, $C_9H_7EtO_2$, is a viscid oil; it has no constant boiling point, but after boiling for a long time it distils at 264—270°, and condenses to a colourless, mobile oil. The *methyl* salt, $C_9H_7MeO_2$, crystallises from dilute alcohol in prisms or needles, melts at 76°, and is readily soluble in all solvents except water. A vapour-density determination in anthracene vapour showed that it is at first polymeric, but, the transformation into simple molecules takes place much more quickly than is the case with the corresponding salt of the γ-acid.

The acids described above are very similar in appearance and solubility to α-isatropic acid (Lossen, *Annalen*, **138**, 235), β-isatropic acid (Fittig, *Annalen*, **206**, 34), and the isatropic acids which the author has previously obtained from atropine, but they are not identical with any of these compounds. β-Isatropic acid seems to be formed when hydrogen chloride is passed into a methyl alcoholic solution of α-isatropic acid. *Methyl β-isatropate* melts at 91°.

The molecular weight of δ-isatropic acid, methyl and ethyl δ-isatropate, and methyl and ethyl γ-isatropate was determined by Raoult's method in glacial acetic acid solution, and in all cases the results agreed for the molecular formulæ given above; the salts are all reprecipitated unchanged when water is added to the glacial acetic acid solution. The acid filtrate from the atropic acids (see above) contains ecgonine and a very small quantity of a complex mixture of acids consisting principally of δ-isatropic acid, but in which benzoic acid was recognised. Anhydroecgonine is also sometimes present. The quantities of ecgonine and isatropic acids are approximately in accordance with the equation given above.

Boiling baryta decomposes the amorphous alkaloïd in a similar manner to hydrochloric acid, but the ecgonine is partially decomposed.

Other bases occurring with cocaïne also yield ecgonine when treated with acids. The author has succeeded in preparing benzoylecgonine from ecgonine. F. S. K.

The Berberine Alkaloïds. By E. Schmidt and F. Wilhelm (*Arch. Pharm.* [3], **26**, 329—365; compare Abstr., 1885, 675; 1886, 633, 1041; 1887, 174, 383, 505, 1057).—*Hydrastine.* By Wilhelm.—The extract obtained by treating the coarsely powdered root of *Hydrastis canadensis* with water acidified with acetic acid at 100°, is evaporated to a syrup and excess of dilute sulphuric acid added, when berberine sulphate separates. The filtrate neutralised with ammonia gives a precipitate containing much hydrastine; this is separated, and on adding ammonia in excess to the filtrate a further precipitate is produced, which contains *canadine*. Both precipitates boiled with ethyl acetate give solutions which on cooling deposit hydrastine in large crystals, somewhat coloured, but rendered pure by recrystallisation. The crystals from the second ammonia precipitate are much purer than those from the first; by slow evaporation of the ethyl acetate solution they can be obtained as large as walnuts. *Hydrastine picrate*, $C_{21}H_{21}NO_6,C_6H_2(NO_2)_3\cdot OH + 4H_2O$, is thrown down as an amorphous, yellow precipitate, which is deposited from its boiling alcoholic solution in splendid, yellow needles. By the action of ethyl iodide on hydrastine under pressure, a well-crystallised ethiodide,

$$C_{21}H_{21},NO_6,EtI,$$

can be obtained of the form $\infty P.\infty P\infty.\breve{P}\infty.(P.\breve{P}\infty)$, melting at 205° to 206°. The corresponding chloride could only be obtained in a gummy mass; its solution was therefore precipitated with platinum and gold chlorides respectively, and the corresponding double salts were obtained and analysed. Both are amorphous, the platinochloride being light red, melting at 207°, and having the composition

$$(C_{21}H_{21}NO_6,EtCl)_2PtCl_4,$$

and the aurochloride being yellow, melting about 110°, and having the composition $C_{21}H_{21}NO_6,EtCl,AuCl_3$. *Hydrastine-ethylammonium hydroxide*, obtained by exactly precipitating the iodine from hydrastine ethiodide by means of silver oxide, concentrating the filtrate, and

allowing to remain over sulphuric acid, appears as compact, slightly coloured crystals, which are purified by recrystallisation from hot water. Their composition is $C_{21}H_{21}NO_6Et{\cdot}OH$, showing that the hydrastine has the character of a tertiary base, and does not, as Power supposed, belong to the imido-bases. The attempts to obtain a hydro-compound by the action of nascent hydrogen on hydrastine, both in acid and alkaline solution, were unsuccessful. The evidence as to the existence of a third alkaloïd, canadine, was doubtful.

Action of Oxidising Agents on Hydrastine. By E. Schmidt and F. Wilhelm.—Hydrastine, when treated with manganese dioxide and sulphuric acid, yields opianic acid and *hydrastinine*. Oxidation with platinic chloride gives the same products. Potassium permanganate in alkaline solution produces hemipinic and nicotinic acids; in acid solution opianic acid is produced, as one of the authors had ascertained when Freünd and Will's publication of the same fact first appeared. The base formed simultaneously was not isolated, but by employing barium permanganate hydrastinine in small quantity was obtained along with opianic acid. Chromic acid yielded the same two products.

Comparing narcotine and hydrastine, E. Schmidt considers that the former contains three methoxyl-groups, thus: $C_{19}H_{14}(OMe)_3NO_4$, whilst the latter contains only two, thus: $C_{19}H_{15}(OMe)_2NO_4$. Since the oxidation of narcotine with manganese dioxide and sulphuric acid yields opianic acid and cotarnine, and under the same conditions hydrastinine gives opianic acid and hydrastinine; further, as opianic acid contains two methoxyl-groups, and cotarnine contains one of these groups, as shown by Wright, it follows that hydrastinine contains no methoxyl-group, and cotarnine may prove to be a methylated hydrastinine. The author hopes later to succeed in converting hydrastine into narcotine. J. T.

The Acids of Pig's Bile. By S. Jolin (*Zeit. physiol. Chem.*, **12**, 512—557).—In a former paper (see Abstr., 1887, 742) it was shown that in pig's bile there is, in addition to Strecker's hyoglycocholic acid, a second acid called β-hyoglycocholic acid. These two acids can be separated by fractional saturation with sodium sulphate of the bile from which the mucin has been removed. In the present paper, after a review of previous researches on the subject, the method of preparation of the bile and separation of the two acids is described in detail. The α-acid (Strecker's) is comparatively easily freed from impurities; but the β-acid and its salts are much more difficult to obtain pure. After a complicated process of solution and reprecipitation, the sodium salt of the β-acid was prepared fairly free from colouring matter; a crystalline substance of doubtful nature was next separated; and finally a number of only partially successful efforts were made to free it from admixture with hyotaurocholic acid, and small quantities of the α-acid or its salts. There are a large number of elementary analyses given of this compound in different stages of its purification; all attempts to prepare a crystalline compound from it were unsuccessful. The concluding pages of the paper give a list of the reactions of the acid, and of a number of its metallic salts, but

the value of such observations seems to be considerably discounted by the fact that the material under investigation is admitted to be impure. W. D. H.

Lecithin. By E. Gilson (*Zeit. physiol. Chem.*, **12**, 585—602).—Diaconoff (*Centr. Med. Wiss.*, 1868) regarded lecithin as a compound of the nature of a salt of neurine, which played the part of the base with distearylglycerophosphoric acid. Strecker (*Annalen*, 1868), on the other hand, considered lecithin as an ether-like combination, the neurine being united to the acid by means of the oxygen of the hydroxyl. In favour of the latter view, Hundeshagen has stated that the neurine salt of distearylglycerophosphoric acid prepared synthetically has none of the properties of lecithin.

In the present research, lecithin was prepared with great care from yolk of egg, and the decomposition-products of the action on it of sulphuric acid of strengths varying from 10 to 50 per cent., and also of dilute alkalis (1 per 1000 to 5 per cent. sodium hydroxide solution), were investigated. It was found that sulphuric acid acts exceedingly slowly in decomposing lecithin, especially when the acid employed is of the lesser strength mentioned above. The products of the action are small quantities of glycerophosphoric acid, another phosphorus-containing substance (distearylglycerophosphoric acid?), and free phosphoric acid; the last-named is the most abundant. Dilute alkalis act more energetically than the acid; the chief product is glycerophosphoric acid.

These results certainly negative Diaconoff's theory that neurine plays the part of a base in a combination resembling a salt, and the conclusion is drawn that Strecker's is the correct view to take of the nature of lecithin. W. D. H.

Physiological Chemistry.

Tension of Oxygen in the Blood, and in Solutions of Oxyhæmoglobin. By G. Hüfner (*Zeit. physiol. Chem.*, **12**, 568—584).—The part played by the partial pressure of oxygen in causing the dissociation of oxyhæmoglobin at the normal body temperature, is a problem which has not been fully worked out. Paul Bert has shown that the dissociation occurs more easily at 40° than at temperatures below that point. Frankel and Geppert obtained similar results, and the author has previously shown (*Zeit. physiol. Chem.*, **6**, 94) that besides pressure and temperature, another very important factor is the concentration of the solution of oxyhæmoglobin used. The present research was undertaken not only with fresh defibrinated blood of the ox and dog, but also with solutions of oxyhæmoglobin crystals from the ox, which are more readily obtained in larger quantities, and are also much more soluble than the dog's oxyhæmoglobin previously used. The apparatus used is fully described and figured. It consists of the

following parts:—(1.) A bulb apparatus, in which the blood or oxyhæmoglobin solution and the gases to be investigated can be collected separately, and subsequently mixed and shaken vigorously. (2.) A manometer by which the pressure of the gases before and after the admixture can be read off. There are also means by which the gases can be analysed; mixtures of oxygen and nitrogen were those always employed. (3.) The whole apparatus is immersed in a very large water-bath, and thus the temperature at which all the processes are carried out can be regulated and observed. Barometrical readings are also necessary for correcting the various readings.

A number of observations at 20° and 35° gave results which demonstrated that dissociation occurs more readily at the latter temperature than at the former. The following may be taken as examples:—

Partial Pressure of Oxygen in millimetres of Mercury.

	At 20°.		At 35°.	
	Before shaking.	After shaking.	Before shaking.	After shaking.
1	42·17	43·15	42·38	46·51
2	46·27	46·63	46·31	50·73
3	54·77	56·26	54·73	57·49

A similar table shows also that the volume of oxygen was increased by the vigorous admixture of the mixed gases with the solution of oxyhæmoglobin or blood to a much greater extent at 35° than at 20°.

Another series of observations in which the temperature remained approximately constant at 35°, and in which the pressure was varied, showed that at this temperature (34—35°) the limit was reached when dissociation of oxyhæmoglobin no longer took place, on increasing the partial pressure of oxygen to 62—82 mm. of mercury; this corresponds with about 300 mm. of atmospheric pressure.

The same figure approximately was obtained whether the oxyhæmoglobin was used in a pure condition dissolved in a 0·1 per cent. solution of sodium carbonate, or whether fresh defibrinated blood was employed. The solution in the former case contained 6 to 8 per cent. of oxyhæmoglobin.

One would naturally expect that at higher temperatures, for instance in the blood during fever, the conditions under which oxyhæmoglobin is dissociated would be different; but for healthy, warm-blooded animals, a fall in atmospheric pressure to below 300 mm. of mercury would be for physico-chemical reasons exceedingly perilous; such a pressure would be attained at a height of 5500 metres.

W. D. H.

Uric Acid in the Urine of Herbivora. By F. Mittelbach (*Zeit. physiol. Chem.*, **12**, 463—466).—No data existing as to the occurrence of uric acid in the urine of herbivorous animals, a number of specimens

of such urine were examined with the following results, Ludwig's method of separating uric acid being employed.

Urine of Oxen.—Twenty-three specimens were examined; they varied in sp. gr. from 1·033 to 1·0425, and as a rule the reaction was strongly alkaline. Phosphoric acid was present in 12, and absent or nearly so in 11 cases. Uric acid was also present; in six cases in which a quantitative analysis was made, the quantity present varied from 8 to 45 milligrams per 100 c.c. of urine. In the urine of seven cows, the results were similar; the sp. gr. was, however, less (1·005—1·011).

Urine of Sheep.—Seven specimens were examined; the sp. gr. varied from 1·010 to 1·0425; the reaction was alkaline; uric acid was present; phosphoric acid was present in traces in two cases only.

Urine of Horses.—Five specimens were examined; all gave an abundant sediment of calcium carbonate; all were strongly alkaline, and had an average sp. gr. of 1·045. Uric acid was present, and phosphoric acid absent in all the specimens.

Urine of Pigs.—Nine specimens were examined; the results were somewhat different from the preceding, the pig being omnivorous; the reaction was acid; the sp. gr. 1·006—1·032; phosphoric acid was present in all cases; so also was uric acid (3—30 milligrams per 100 c.c. of urine). W. D. H.

Influence of Atropine on Salivary Secretion. By J. N. Langley (*J. Physiol.*, **9**, 55—64).—Atropine when administered to animals, causes a stoppage of the secretion of saliva; stimulation of the nerves going to the glands no longer causes the formation of saliva to take place. In a normal condition, stimulation of different nerves causes the formation of saliva of different kinds, differing, that is to say, in the proportion of solids to water. From this and similar experiments it has been concluded that the different nerve-fibres have different functions, and the names, secretory, trophic, and anabolic have been applied to these. In the present research, the cat was the animal experimented on, and it was found by the use of small doses of atropine administered succesively that all varieties of nerve-fibre were equally and simultaneously paralysed. In other words, the phenomena of atropine poisoning give no indication of the existence of more than one kind of secretory nerve-fibre in the chorda tympani. It is shown that this result is not irreconcilable with the conclusions arrived at by Bayliss and Bradford (*Proc. Roy. Soc.*, **40**, 203), with regard to the electrical phenomena which accompany secretion in the submaxillary gland. W. D. H.

Physiological Action of Borneol. By R. Stockman (*J. Physiol.*, **9**, 65—91).—The substances investigated were Borneo camphor, Ngai camphor, and borneol prepared artificially from oil of turpentine. These were contrasted in their action with ordinary or laurel camphor, and with menthol or peppermint camphor.

The three substances first named are identical in chemical composition, and are generally known under the common name of borneol, but they differ considerably in certain physical properties. The

most striking difference is their action on polarised light; an alcoholic solution of Borneo camphor is dextorotatory, that of Ngai camphor lævorotatory, whilst that of borneol artificially prepared has no action at all on polarised light. The formula of borneol is $C_{10}H_{18}O$, a monatomic alcohol of which common camphor, $C_{10}H_{16}O$, is the aldehyde.

All three varieties of borneol produce the same physiological effects. A very complete investigation was made of their action on the different systems of organs in frogs and in mammals, and complete details of the experiments are given. The following are the general conclusions that are drawn: that laurel camphor, borneol, and menthol form a group of substances very closely allied to each other in physiological action. To these may be added camphor monobromide, $C_{10}H_{15}OBr$, a derivative of laurel camphor, the action of which seems to resemble that of borneol more nearly than that of the other two substances. They are all closely related to the alcohol group in their physiological effects, menthol approaching the latter most nearly, but as the number of hydrogen-atoms diminishes, there is an increased tendency to produce convulsions of cerebral origin. It is in the nervous system indeed that the most marked effects of these drugs are manifested; in mammalia, the symptoms are almost entirely cerebral, the spinal cord being affected much less profoundly; in frogs, on the other hand, the brain is affected first, but subsequently the cord, and lastly the motor nerves are paralysed. Camphor and the essential oils have long been used therapeutically in conditions of increased spinal excitability, and this coincides with the results of pharmacological investigation. As cardiac stimulants, the *modus operandi* of these drugs is again closely related to that of alcohol. Borneol and menthol have, however, a marked effect in dilating peripheral vessels, which ethyl alcohol has not. Pure alcohol is not, however, used therapeutically, but mixed with different ethers in the form of wines and spirits, which dilate the vessels; the resemblance, therefore, between ordinary alcoholic stimulants and the camphor-group of drugs in their action on the circulation is very close.

The Chinese place an exaggerated value on Borneo camphor, its price being about £5 per lb., but whether this is due to a superstitious belief in its virtues, or to any real advantage it has over ordinary camphor is difficult to determine, and can only be decided by long clinical experience. Pharmacological experiments certainly do not show that it is markedly different in its action from the other substances of the same group. It is certainly less irritating locally than laurel camphor, and can be given in much larger doses than the latter without causing untoward cerebral symptoms. W. D. H.

Action of Caffeïne and Theïne on Voluntary Muscle. By T. L. Brunton and J. T. Cash (*J. Physiol.*, **9**, 112—136). This paper gives a complete and detailed account of experiments of which a short notice has already appeared (see Abstr., 1887, 985).

W. D. H.

Strychnine Poisoning. By R. W. Lovett (*J. Physiol.*, **9**, 99—111).—There is a marked affinity on the part of certain drugs for

certain organs, or rather a power of certain organs to select some substance from the blood, and by destroying or excreting it, or storing it up, to remove it from the circulation. In the case of strychnine, the question arises, does it exercise its peculiar power on the spinal cord because there is more of the drug present there than in the other organs, or because the cord is more susceptible to its action than muscle, brain, liver, &c.? In attempting to find an answer to this question, the line of work adopted was as follows:—First, by the injection of known amounts of strychnine into frogs of known weight, and by the observation of the time in which convulsions occurred, to construct a table showing the time that various amounts of strychnine require to produce convulsions in frogs of known weight. The table thus constructed may be briefly given thus:—

Dose.	Time between injection of drug and convulsions, in minutes per gram of frog weight.
0·0648 mgrm.	0·657, average of 13 experiments.
0·0324 „	1·0 „ 17 „
0·0216 „	1·5 „ 16 „
0·0162 „	2·05 „ 8 „
0·013 „	2·54 „ 9 „

The second step in the investigation was to take equal weights of the tissues (liver, muscle, spinal cord, blood, &c.) of frogs poisoned by strychnine, to rub each up in a mortar with water, and then to inject the tissue thus finely subdivided, by means of a hypodermic syringe with coarse needle, into a second set of frogs, which were again of known weight. By this means, it was found that injection of the crushed spinal cord rarely failed to produce convulsions, whilst that of the other organs either produced no convulsions at all, or only after a long interval.

Thirdly, by watching the time that convulsions were in appearing, and by knowing the weight of the frogs used, it was possible by consulting the table already given to determine approximately the quantity of strychnine in equal weights of the various tissues of the poisoned frogs. These results are given in tables, but the general conclusion to be drawn from them is that the spinal cord has the power of selecting strychnine from the circulation and of storing it up in its structure. This may be of importance in medico-legal cases, as it shows the best place in which to search for the drug in cases of poisoning where the stomach-contents are not available.

W. D. H.

Chemistry of Vegetable Physiology and Agriculture.

Saccharomyces Apiculatus. By C. Amthor (*Zeit. physiol. Chem.*, **12**, 558—564).—*Saccharomyces apiculatus* has been studied by

Rees, Pasteur, Engel, Hansen, and others. It has been found in the juices of the grape and of other ripe fruits, also in beer. Hansen, from his observations on *S. cerevisiæ* and *S. ellipsoideus*, concludes that different varieties of these species exist. This conclusion is supported by the fact that different yeasts perform different kinds of chemical work in the same nutritive solution (see this vol., p. 184). From the present research, a similar conclusion is drawn with regard to *S. apiculatus*. The subject is not studied from the morphological standpoint, but from the point of view of difference in chemical action.

Must from ripe, fresh grapes was sterilised and analysed: it had the following composition:—

Sp. gr. at 15°	1·08234
In 100 c.c. at 15°—	
Extract (Schultze's method)	21·87
Invert-sugar	20·7216
Acid	1·0125
Fixed acid	1·0083
Volatile acid	0·0033
Nitrogen	0·0552
Colour intensity (Stammer's colorimeter)	4·17

Some of this was divided into two parts; to one part was added a cell of *S. apiculatus* from Niedersaulheim, and to the other, one from Heilbronn. In some weeks' time, when the fermentation had subsided, the two liquids were analysed, with the following results:—

In 100 c.c. at 15°.	Niedersaulheim.	Heilbronn.
Alcohol, vol. per cent.	3·25	4·56
„ wt. „	2·58	3·65
Glycerol	0·31	0·35
Extract (Schultze)	16·09	13·80
Invert-sugar	14·23	11·20
Acid	1·06	1·01
Volatile acid (as acetic acid)	0·13	0·11
Ash	0·19	0·19
Phosphoric acid	0·03	0·03
Nitrogen	0·04	0·04
Sp. gr.	1·06017	1·05199
Colour intensity	2·5	1·89

The marked differences in the composition of the two wines certainly supports the contention that varieties of the yeast employed exist. If the total acid be taken into account, that is, in the solid residue as well as that in the fluids above mentioned, it is found that the total acid formed during fermentation is about three times greater than that found by Pasteur with ordinary yeast. The volatile acid contains but little acetic acid; it smells like coumaric acid, but was not further investigated.

In beer wort, *S. apiculatus* (Heilbronn), caused in 30 days the formation of only 0·93 per cent. of alcohol. Hansen obtained a similar result, and supposed that this form of yeast never produces more than 1 per cent. of alcohol. Jörgensen, on the other hand, considered that the reason lies in the fact that *S. apiculatus* does not ferment maltose.

Some experiments in the present research support the latter contention. Some of the wort was heated with sulphuric acid to convert the maltose into dextrose; the acid was then neutralised with calcium carbonate, and the liquid evaporated to its original volume, sterilised, and to it was added once more a cell of the *S. apiculatus*. After eight days' fermentation the percentage composition of the liquid was—

Alcohol by volume	2·62
„ by weight	2·11
Acid	0·2625
Volatile acid	0·0618

That is, in a third of the time there was about three times as much alcohol formed as before treatment with sulphuric acid.

The large amount of acidity corresponds with what was observed in wine. If *S. apiculatus* does not ferment maltose, the small quantity of alcohol formed in the first case must have been derived from dextrose. Musculus and Gruber (*Zeit. physiol. Chem.*, **2**, 181) show that when diastase acts on starch it produces small quantities of dextrose in addition to dextrin and maltose.

The property of S. *apiculatus* just referred to furnishes us with a means not only of detecting small quantities of dextrose in presence of maltose, but of estimating the quantity present by the amount of alcohol formed. W. D. H.

Changes occurring in Timothy Grass (Phleum pratense). By E. F. Ladd (*Bied. Centr.*, 1888, 574).—Four periods of growth were selected in which to examine the composition of Timothy grass; it was found that the percentage of water diminishes rapidly after blossoming, as does also the woody fibre. As ripening approaches, sugar decreases and starch increases, and the albuminoïds are less digestible during the later periods of growth. In the period between blossoming and perfection of the seed, albuminoïds are converted into amides, either during the migration of albuminoïds from the stem into the seed, or else in conjunction with the migration and conversion of sugar and starch. Consequently, when required for fodder, it is advisable to mow the grass at the period of blossoming.

E. W. P.

Does Grain contain Sugar? By A. v. Asboth (*Bied. Centr.*, 1888, 574).—The author contends that the methods employed by Schlösing, König, and others are incorrect, that sugar is not contained in grain, and that in those cases where sugar has been found, it has been produced by the method employed for its detection.

E. W. P.

The Constituents of Acorus Calamus. By H. KUNZ (*Arch. Pharm.* [3], **26**, 529—536; compare Abstract, 1886, 895).—Calamin, which was discovered by Thoms, is stated in a more recent publication to be identical with methylamine. Guthzeit has also announced the discovery of methyl alcohol in the aqueous distillate from calamus. Thus we have ethereal oil, acorin, methylamine, and methyl alcohol given as integral constituents of calamus; it still remains to be ascertained which are primary and which secondary constituents. From Thoms' investigation it would appear that the ethereal oil is a decomposition-product of acorin. This author also remarks that the methyl alcohol is derived from the methylamine. During Thoms' separation of calamin, he perceived a distinct, herring-like odour which is characteristic of trimethylamine; this induced the author of the present communication to search for choline in calamus, in which he was successful, as shown by the production of its hydrochloride and the platinochloride, $(C_5H_{13}NO)_2,H_2PtCl_6$. The author further proves the absence of methylamine hydrochloride in the salt residue in which Thoms claimed to have found it, whilst trimethylamine and ammonia were present. J. T.

Constituents of Lupin Seeds. By G. BAUMERT (*Arch. Pharm.* [3], **26**, 433—440).—The author gives a summary of the results obtained on this subject during the past 30 years, and appends a list of works and papers published. There are great differences in the composition of seeds of different varieties, thus König gives the mean of 14 analyses of *L. luteus* as: Nitrogenous substances 36·52, fat 4·92, ash 4·04, cellulose 14·04, nitrogen-free extract 27·60; and the mean of 8 analyses of *L. augustifolius* as: Nitrogenous substances 25·37, fat 4·90, ash 3·02, cellulose 12·40, nitrogen-free extract 41·68. It is also known that the various seeds do not all contain the same alkaloïds; the constituents of the yellow lupin (*luteus*) seeds are best known. The aqueous extract of lupin seed reacts strongly acid, this being due mainly to the presence of citric acid and a little malic acid, whilst the presence of oxalic acid is somewhat doubtful. The first two acids amount to about 1·92 per cent. of the dry substance. There is less acid in the seeds of the blue lupin (*angustifolius*). Besides the yellow liquid fat extracted by ether, Beyer found a wax-like compound soluble in warm alcohol; both fats contain phosphorus.

The liquid fat contained	C 75·700,	H 11·350,	P 0·098,	O 12·852,	Beyer.
The wax-like fat contained.....	C 72·68,	H 10·84,	P 1·56,	O 14·92,	,,
Lupin fat.....	C 75·94,	H 11·59,	—	O 12·47,	König.

With concentrated sulphuric acid, the powdered seed gives a red coloration due to a cholesterin, which, according to Schulze and Barbieri, has the same crystalline form as ordinary cholesterin, but melts at 136° to 137° instead of at 145°; its specific rotatory power is $[\alpha]_D = -36·4$. Of carbohydrates neither starch nor inulin occurs according to Ludwig, and sugar is only found as a decomposition product; a dextrin-like substance with dextrorotatory power has been described;

this by the action of mineral acids produced a sugar capable of reducing Fehling's solution. Steiger has found this to be a white hygroscopic powder of the composition $C_6H_{10}O_5$, easily soluble in water, almost insoluble in alcohol and ether. Its aqueous solution is not coloured by iodine, diastase has no action on it, nitric acid converts it into mucic acid, and by treatment with acetic anhydride it takes up three acetyl-groups. The product, therefore, is not glucose but is identical with galactose from milk-sugar, hence the carbohydrate from *L. luteus* is named galactan, and considered as the β-compound to distinguish it from α-galactan, Muntz's galactin obtained from lucerne. Schulze and Steiger obtained another compound from the same variety also insoluble in water, and convertible into galactose by the action of boiling acids. This so-called paragalactin has the formula $C_6H_{10}O_5$, and yields a triacetyl-compound which decomposes at 225° without melting, whilst that from β-galactan melts at 101—102°. Besides the above carbohydrates, cellulose to the extent of 12 to 14 per cent. occurs in lupin seeds. The albumin of these seeds consists mainly of conglutin according to Ritthausen, along with small quantities of legumin and a little vegetable albumin. Conglutin and legumin differ in their solubility in salt solution; the former contains 18·67 per cent. nitrogen, and the latter 17·41. All lupins probably contain several alkaloïds, amounting to 0·04 to 0·81 per cent. The author has found in yellow lupin the crystallisable alkaloïd lupinine, $C_{21}H_{40}N_2O_2$, and the liquid lupinidine, $C_8H_{15}N$. In the blue lupin only a liquid alkaloïd has been investigated which Hagen has named lupanine; its composition is $C_{15}H_{25}N_2O$. Campani has obtained yet another alkaloïd from the seeds of *L. albus*.

Other nitrogen compounds of the amide-group have not been isolated from the ungerminated seed; but after germination a series of compounds produced by the decomposition of the albumin has been obtained by Schulze, Barbieri, and Steiger: asparagine is the principal product, and there are besides phenylamidopropionic acid, amidovaleric acid, leucine, tyrosine, xanthine, hypoxanthine, lecithin, peptone, and arginine, $C_6H_{14}N_4O_2$, a base analogous to creatinine. Choline also was detected.

Schulze and Barbieri found a new glycoside in *L. luteus* of the composition $C_{29}H_{32}O_{16}$ which they named lupinin, but it is better called lupiniin to distinguish it from the similarly named alkaloïd. It crystallises in fine needles, dissolves in alkaline solutions with a yellow colour, and on treatment with acids takes up 2 mols. H_2O with the formation of lupigenin, $C_{17}H_{12}O_6$, and glucose. Hot water also produces the reaction. J. T.

Influence of a Crop or Covering on the Physical Characters of a Soil. By E. Wollny (*Bied. Centr.*, 1888, 505—524).—In continuation of his previous experiments (Abstr., 1884, 922), the author has investigated the effect on the amount of water when the soil is bare or covered with vegetation, &c. He finds that soil covered by vegetation is drier than if it were bare and unshaded, whilst if it be covered by straw, farmyard manure, &c., it is moister during warm

weather than if it were bare, the moisture present is higher the thicker the covering, and it is driest when there is a covering of live vegetation. It is consequently an error to suppose that land is kept moist by being shaded by a crop of potatoes, peas, &c., the contrary being the case, and is occasioned by the shelter which the plants afford to the soil, the rain remaining on their leaves and there evaporating, further, the dry condition is assisted by the transpiration of water by the leaves themselves. The dry condition of the surface is also dependent on the activity of growth of the plants and their close standing; but when the surface is covered by any dead matter, then the state of affairs is reversed, and the soil is the moister the thicker the covering; the amount of water in a bare uncovered soil is a mean of the other two quantities. As regards the amount of drainage water, it is found that a greater quantity passes into the deeper strata when the soil is bare than when it is covered, and a greater quantity percolates through a soil covered by straw, &c., than through a perfectly bare soil. The character of the plants growing also affects the amount of drainage, unmanured allowing of more drainage than manured plants, and when vegetation is thick and strong there is but little drainage; the covering of dead material only increases the drainage so long as it is not more than about 5 cm. thick, beyond that drainage is diminished. Full accounts of the experiments made and tables of results are given. E. W. P.

Manuring of Clover. By J. Samek (*Bied. Centr.*, 1888, 527—529).—On sandy loam, clover was grown and manured with various manures, including basic slag. The least effect on the first cut was produced by the slag, whilst the third cut was heavier than that produced by any of the other manures. Gypsum had no effect. The increase produced by potash salts was neutralised by the extra expense incurred. Of nitrogenous manures, blood-meal mixed with superphosphate was found to be the best. E. W. P.

Manuring with Nitre. By M. Fleischer (*Bied. Centr.*, 1885, 525—527).—White lupines, clover, potatoes, &c., were manured with a mixture of kainite, superphosphate and Chili saltpetre, the latter in increasing quantities. An increased yield of the leguminous plants was obtained, and the increase more than paid for the extra expenses. The land was light. E. W. P.

Basic Slag as a Manure for Oats. By A. Emmerling (*Bied. Centr.*, 1888, 529—531).—In the dry season of 1887, and on a loamy soil, excellent results were obtained from a mixture of basic slag and Chili saltpetre in the proportion of 8 : 3. The superiority of basic slag over superphosphate is accounted for in this year, by the action of superphosphate not being brought out fully unless there is a fair supply of moisture. E. W. P.

Fossil Milk. By V. Storch (*Bied. Centr.*, 1888, 566—567).—This so-called fossil milk, which occurs as a white, porous mass in Bergthorshvol in Iceland, contains more phosphoric acid than is found in

coprolites, &c., the proportion of lime and phosphoric acid present closely resembling that found in the coagulum of milk in use in Iceland, and called "Skyr," which is produced by the combined action of rennet and sour milk. Organic matter has been detected, which reacts with tungstic acid, tannin, and iodine, but which produces no results with Millon's reagent and does not give the biuret reaction. The best sample contained 11 to 12 per cent. combustible matter, whilst the pure organic matter contained 10 per cent. of nitrogen. Under the microscope, a tissue consisting of pyrenomycetes was observed. E. W. P.

Analytical Chemistry.

Testing Potassium Carbonate. By E. Bohlig (*Arch. Pharm.* [3], **26**, 541—542).—To avoid error in testing potassium carbonate by means of silver nitrate solution, the author recommends the following method of procedure:—

Pour gradually a solution of 0·5 gram of potassium carbonate into a solution of 3 grams of silver nitrate in 100 c.c. of distilled water. The precipitate should be pure white, not yellowish. A further test may be made by dropping silver nitrate solution not in excess into the carbonate solution. If the precipitate is now white, the carbonate also contains hydrogen carbonate. J. T.

Action of Iodine on Hydrogen Arsenide and Antimonide. By O. Brunn (*Ber.*, **21**, 2546—2549).—Hydrogen arsenide present in hydrogen sulphide can be determined by passing the mixture through a tube containing iodine. The contents of the tube are afterwards washed into a glass, hydrogen sulphide free from hydrogen arsenide passed through, and the arsenic sulphide so formed is determined as ammonium magnesium arsenate, which is dried at 102—103° and weighed. Some results obtained by the method are given, as well as results obtained with the same mixture of gases, by passing the gas through a dilute solution of silver nitrate, and determining the arsenious acid by means of an iodine solution. The results show that the method is trustworthy. Hydrogen antimonide, which behaves towards iodine similarly to the arsenide, can also be readily determined by this method. N. H. M.

Detection of Poisoning by Caustic Alkalis. By Vitali (*Arch. Pharm.* [3], **26**, 516; from *L'Orosi*, 1888, 37).—Detection is difficult, as the alkali quickly becomes converted into carbonate or it combines with albuminoïd substances. If solid matter such as flesh is to be examined, it is finely divided and digested for 24 hours with 8 volumes of alcohol, whilst liquids are evaporated to dryness at the lowest possible temperature, and with a minimum amount of ex-

posure to the air. A little of the alcoholic extract is shaken with mercurous chloride, which blackens in presence of caustic alkalis whether free or combined with proteïd substances. The black powder should disappear on digestion with nitric acid, otherwise it may be due to sulphur compounds. If a positive result has been obtained, the remainder of the alcoholic solution is treated with sulphuric acid when the alkaline sulphate is precipitated. The precipitate is dried and ignited, dissolved in a little water, and a portion is used to obtain crystals by evaporation, whilst the remainder is employed to determine whether sodium or potassium is in question. This gives only the free alkali: to obtain the alkali combined with albuminoïd compound, the residue from the original alcoholic extract is extracted with boiling water, to this solution, after being brought to a syrup, is added 6 volumes of absolute alcohol, and then sufficient ether to precipitate all that will come down. If the precipitate blackens mercurous chloride, caustic alkali combined with albuminoïd matter is indicated. The nature of the alkali is determined by igniting the precipitate, dissolving in hydrochloric acid and examining further. Finally, the alkali in combination with proteïd substances insoluble in water is obtained by extracting the flesh residue insoluble in alcohol and water with dilute hydrochloric acid and proceeding as above.

J. T.

Haycraft's Method of Estimating Uric Acid in Urine. By A. Herrmann (*Zeit. physiol. Chem.*, **12**, 496—501).—Some modifications in the details of Haycraft's method of estimating uric acid in urine (*Brit. Med. Jour.*, **2**, 1885, 1100) are suggested, and its accuracy tested in comparison with Ludwig's method (*Wiener Med. Jahrbücher*, 1884). It was found that on the average, the results by Haycraft's method were 0·0029 gram per 100 c.c. of urine (or 7·9 per cent. of the total uric acid) higher than by Ludwig's method. With solutions of pure uric acid, there is by both methods a loss of 2 per cent., and the explanation of the difference probably is, that the ammoniacal silver solution used by Haycraft precipitates other substances from the urine; for instance, those of the xanthin-group in addition to uric acid.

The occurrence of sugar or albumin in the urine does not at all influence the reactions on which Haycraft's method depends. This is one great advantage of the method; another is the ease and rapidity with which the analysis can be made, and for a series of comparative clinical observations it is a very suitable method indeed.

It is pointed out that the addition of sodium hydrogen carbonate to the urine does not entirely prevent the reduction of the silver as stated by Haycraft; but that the reduction is so small in the time occupied by the analysis, especially if the filter-pump is used, that it may be disregarded.

W. D. H.

Estimation of Uric Acid in Urine. By F. Czapek (*Zeit. physiol. Chem.*, **12**, 502—511).—This method claims to be even simpler than Haycraft's, of which it is a modification (see preceding Abstract), as it does away with the use of the filter-pump. It, however, like Haycraft's, gives too high results when tested with urine, and the delicacy of the two tests is about equal.

With pure uric acid in solution, it is stated to give results equally as good as those obtained by Ludwig's method by direct estimation. This method is recommended for use in clinical work. The essential feature is that a known quantity of ammoniacal silver solution is added to the urine, the uric acid being thus precipitated as silver urate; this is filtered off, and the silver is estimated not in the precipitate (as in Haycraft's method), but in a certain measured proportion of the filtrate. From these data, the amount of silver residue is obtained, and thus the amount of silver and uric acid in the precipitate. By this process not only is the use of the filter-pump unnecessary, but it also does away with the washing of the precipitated urate which is very troublesome. The silver in the filtrate is estimated by warming and titrating with potassium sulphide or sodium sulphide solution, the browning of lead paper being the indicator used for ascertaining the termination of the reaction. Several successive titrations are necessary to obtain a correct result. W. D. H.

Densimetric Estimation of Proteïds. By HUPPERT and ZÁHOŘ (*Zeit. physiol. Chem.*, **12**, 467—483).—Methods have been devised for the quantitative estimation of proteïds in solution by means of multiplying the loss in specific gravity which such solutions undergo on the removal of the proteïd, by a constant factor, under the mistaken impression that the loss of density is directly proportional to the amount of proteïd removed. In the case of grape-sugar where a similar method has been adopted, Budde (this vol., p. 198) has shown that from its very nature the factor is a variable one; and the first part of the present paper is devoted to an algebraical demonstration that the same is the case with albumin in solution. For the complete exposition of this point, the original paper must be consulted. The essential facts are, however, these: the sp. gr. of a liquid is its weight divided by its volume; and its volume may be obtained by dividing its weight by its sp. gr. Let x = grams of proteïd in 100 grams of solution:

Let v = sp. gr. of the solution,
v_2 = ,, of the proteïd,
v_1 = ,, of solution *minus* proteïd.

The value of v_1 may be calculated; it is the weight of the proteïd-free liquid divided by its volume, which is the volume of the original liquid, $\frac{100}{v}$ *minus* the volume of the proteïd removed $\frac{x}{v_2}$: therefore

$$v_1 = \frac{100 - x}{\frac{100}{v} - \frac{x}{v_2}};$$

this gives as the value of x—

$$x = \frac{100\,v_2}{v(v_2 - v_1)}\,(v - v_1),$$

$v - v_1$ is the loss of sp. gr., and the fraction $\frac{100v_2}{v(v_2 - v_1)}$ is the sup-

posed constant factor: that it cannot be constant is seen from the fact that v and v_1 are both variable quantities. Experiment also showed that v_2, the sp. gr. of the contained proteïd, is likewise variable. If x, the percentage of proteïd, is known, v_2 can be calculated from the same formula as follows:—

$$v_2 = \frac{xvv_1}{xv - 100(v - v_1)}.$$

A series of 20 estimations of the amount of proteïd coagulable by boiling after adding acid was made. The value x was determined by weighing the proteïd precipitate and the specific gravities by Sprengel's pycnometer. The solutions employed were ascitic fluids, blood plasma, and solution of white of egg, and the value v_2 (sp. gr. of the proteïd) was found to vary between 1·352 and 1·405. The average of the 20 observations gives $v_2 = 1{\cdot}3747$ which may be used in approximate determinations. In connection with the value of v_2 (sp. gr. of the proteïd), it may be noted that it is influenced by the quantity of proteïd and the density of the solvent, v_1. Now it is known that certain salts precipitate proteïds in an unchanged state when the concentration of the salt solution reaches a certain height, salt solutions of small density do not precipitate the proteïd, but they increase its density; when the concentration of the salt in solution is increased, a point is at last reached where the contraction of the proteïd becomes so great that it is precipitated. The concentration of the salt solution will also vary with the salt used.

Tables are then given of comparative experiments in which the quantity of proteïd was determined by weighing, and by calculation from such formulæ as have been instanced; and it is shown that the densimetric method yields only approximate results. W. D. H.

Densimetric Estimation of Albumin in Urine. By H. Záhoř (*Zeit. physiol. Chem.*, **12**, 484—495).—Although it has been shown (see preceding Abstract) that from the theoretical side there is much to be urged against the densimetric method, yet it is found in practice, in cases where accuracy to the first place of decimals is sufficient, that the method is extremely quick and handy. This is felt nowhere so much as in estimations of albumin in urine, and the following method is simple, and can be carried out clinically:—

The filtered urine is mixed with just so much dilute acetic acid that when it is boiled all the albumin is coagulated; the right proportion may be ascertained with a small quantity of the urine in a test-tube beforehand. On being filtered from the coagulum, the filtrate should give no cloudiness with acetic acid and potassium ferrocyanide. A quantity of the urine is then placed in a flask firmly closed with a clean caoutchouc stopper. The flask is hung for 10 to 15 minutes in a large bath, filled with water kept boiling. By this means the albumin is precipitated. It is then filtered off, the funnel leading through a cork with a hole in it into a flask, and being covered with a glass plate. The density of the urine and the filtrate is then estimated, not with a pycnometer (that is unnecessary for clinical work), but with an aräometer marked to four places of decimals. Both fluids

must be kept at the same temperature. This is best done by placing them in two cylinders, both immersed in a large vessel of water, which should be kept at the same temperature if a series of observations are to be made. The temperature of 17·5° will be found most convenient. The difference between the two specific gravities is then multiplied by 400, and the product gives the number of grams of albumin in 100 c.c. of urine.

A large number of illustrative experiments are quoted, in which the approximate accuracy of this simple process is demonstrated. The number 400 is the mean in round numbers of the factor $\frac{100v_2}{v(v_2 - v_1)}$. The question naturally arises why a constant factor should give such good results in albuminous urine, when not only theoretically but also in practice it yields fallacious results in other albuminous fluids, such as the blood, transudations, white of egg, &c. The reason is that the factor must be multiplied by the difference in the specific gravities. In proteïd solutions (other than albuminous urine) this difference varies from 0·0016 and 0·0128, whilst in albuminous urine this difference is much smaller, varying between 0·00012 and 0·0020, that is, in the former case the difference is from six to 13 times greater than in the latter, and therefore so many times greater will be the error introduced by the use of a constant factor. In the case of urine, this error may be neglected. W. D. H.

Biological Test for Malt. By F. Faulkner and W. Virtue (*Bied. Centr.*, 1888, 481—482).—A flask of 100 c.c. capacity is half filled with distilled water, closed with cotton-wool and sterilised; after cooling to the proper temperature for mashing, a teaspoonful of malt is introduced, and the whole is kept for two hours at the mashing temperature on a water-bath, after which it is cooled and placed in a thermostat. After some hours, the upper liquid becomes clear; observations must now be made as to whether the liquid becomes thick, or if gas is evolved, both of which occurrences are signs of decomposition. This process serves to estimate the quality of malt. Comparative experiments must necessarily be made under like conditions; the higher the temperature in the thermostat (with certain limits) the more rapid will be the development of micro-organisms; a concentrated wort decomposes less rapidly than a dilute: if much acid is present, then the wort more readily resists change, consequently the acidity should be reduced before testing; the presence of caramelised matter tends to preserve wort unaltered, so that a dark malt must be carefully examined for caramel. The presence of coombs may retard or advance decomposition, as there is more or less of caramelised compounds present. E. W. P.

General and Physical Chemistry.

University of Michigan

Chromium and Manganese in Fluorescent Mixtures. By L. DE BOISBAUDRAN (*Compt. rend.*, **107**, 311—314).—Calcium carbonate containing chromium oxide, precipitated by ammonia, becomes green when calcined in presence of air, but if it contains a trace of sodium carbonate it becomes yellow. A similar mixture precipitated by sodium carbonate becomes green, however, when strongly heated, if it is previously moistened with hydrochloric acid or ammonium chloride. In a vacuum, the yellow substance shows a green fluorescence, but remains yellow if the discharge is not too intense and its passage is not long continued. If, however, the tube is very gently heated whilst the discharge is passing, the substance becomes green, the change taking place more quickly the smaller the quantity of sodium present. After cooling, the substance shows a green fluorescence.

When the yellow mixture is exposed to the air for some time, it is converted into carbonate without change of colour, but is no longer fluorescent. When gently heated in a vacuum, the colour changes to green, but the mixture remains non-fluorescent. If, however, it is strongly heated in presence of air, it regains its fluorescent properties. A similar mixture containing 5 per cent. of chromium oxide becomes green when heated in a vacuum, and remains green if kept for several months in an open vessel, but then will not fluoresce.

The fluorescence of calcium oxide containing iron or chromium cannot be attributed to the simultaneous presence of small quantities of copper. It would seem, therefore, that both ferric oxide and chromic oxide are active substances with respect to calcium oxide, but their activity is of an order somewhat different from that of other substances. The green in the spectrum of the fluorescence of calcium oxide containing iron or chromium is usually less intense than with calcium oxide alone, but the difference is not sufficient to decide whether the activity of iron and chromium is positive or negative. Whilst, however, they have little effect on the green, they are negatively active to the other rays, the intensity of which is reduced, and hence the colour of the fluorescence becomes green. C. H. B.

Influence of Inactive Substances on the Specific Rotatory Power of Tartaric Acid. By R. PŘIBRAM (*Monatsh.*, **9**, 485—504).—The author confirms the experiments of Landolt (Abstr., 1881, 257), and finds that the specific rotatory power of tartaric acid rapidly diminishes when acetone is added to the aqueous solution. Alcohols behave in a precisely similar manner, those of highest molecular weight producing the greatest change. Solutions originally dextrorotatory may be made lævorotatory by the addition of butyl alcohol; whilst isomeric alcohols, propyl and isopropyl on the one hand, and trimethyl carbinol and isobutyl alcohol on the other, bring about

practically similar changes. Acetic acid also lowers the rotatory power to such an extent as to make it possible to estimate the amount of it present in aqueous solutions by means of tartaric acid.

G. T. M.

Transparency of Metals. By W. WIEN (*Ann. Phys. Chem.* [2], **35**, 48—62).—This research was undertaken with a view of investigating more fully than had hitherto been done, the relation between electrical conductivity and optical transparency in metals, with the object of determining how far they agree with Maxwell's law.

Maxwell's experiments with gold leaf had given a greater transparency relatively to its electrical conductivity than was consistent with the law referred to, but he considered the results open to some doubt owing to defects in the method of observation.

The metals experimented on by the present author were platinum, gold, silver, and iron.

The intensity of the beam of light, both with and without a thin layer of metal interposed, was determined by measuring the change of resistance in a modified form of bolometer forming one of the arms of a Wheatstone bridge. The bolometer was formed of silver foil covered with lampblack to reduce the reflection to a minimum, and was found to be very sensitive, as the temperature coefficient of silver is a high one, and a sufficiently large piece of the thin foil could be employed to intercept all the rays from the source of light, without making the resistance too small for its variations to be determined with accuracy.

The author finds, in the case of the metals experimented with, that the absorption is less, and therefore the transparency greater than is in accordance with Maxwell's law.

Similar results were obtained for the dark rays of the spectrum, showing that there was no better agreement with Maxwell's law in the case of the longer waves, although according to Maxwell's theory, there ought to be a closer agreement in the latter case, as his theoretical law would only be strictly true for waves of infinite length.

G. W. T.

Voltaic Balance. By G. GORE (*Chem. News*, **58**, 64).—Two couples of zinc or magnesium and platinum are immersed simultaneously in two vessels of distilled water, they are then opposed to each other through an ordinary astatic galvanometer of 100 ohms, or through a Thomson's reflecting galvanometer of 3040 ohms resistance, so as to balance one another and cause no deflection; this arrangement is remarkably sensitive in some cases. For example: with a zinc and platinum couple and the astatic galvanometer, 1 of iodine in about 3,520,970 parts of water, or 1 of hydrochloric acid in about 9,388,185, or 1 of chlorine in about 1,300,000,000, causes deflection, whilst with the reflecting galvanometer 1 of hydrochloric acid in about 23,250,000 of water may be detected; on the other hand, with a magnesium and platinum couple, 1 of bromine in about 344,444,444 of water, or 1 of chlorine in about 17,612,000,000, and with the reflecting galvanometer even more dilute solutions upset the equilibrium.

Large quantities of neutral salts are required to disturb the balance.

The degree of sensitiveness varies directly with the affinity, the dissolved substance has for the positive metal, and indirectly with its affinity for the negative metal. D. A. L.

Electrification of a Gas by a Glowing Platinum Wire. By R. Nahrwold (*Ann. Phys. Chem.* [2], **35**, 107—121).—In his former investigations (*ibid.*, **5**, 460, and **31**, 448), the author found that a glowing platinum wire immersed in atmospheric air gives rise to an electrification in the surrounding air, even when the wire itself is not electrified by any external source. He also found that when the wire glows feebly, the electrification is positive, but becomes negative when all dust particles are consumed and the wire is glowing brightly. He came to the conclusion that these results were to be accounted for by the burning of dust particles and other non-gaseous matter in the vicinity of the wire, and by the disintegration of the wire at higher temperatures, and the consequent giving off of charged particles of metal. These conclusions are in conformity with the results obtained in the present paper, which likewise confirm the results of Elster and Geitel (*Ann. Phys. Chem.* [2], **31**, 109), that a feebly glowing platinum wire immersed in atmospheric air becomes negatively charged, but charges the air positively.

When the air was replaced by hydrogen, similar results were obtained when the gas contained dust particles, or when the wire was not perfectly clean, but at higher temperatures, although a negative charge was obtained, it was much less than with atmospheric air, and at the same time it was definitely proved that the disintegration of the wire is very small in hydrogen compared with its amount in atmospheric air. A curious result was observed when the wire, after exposure to the air, was heated in an atmosphere of hydrogen, namely, it was destroyed when a dull red heat was attained. This did not occur when the wire was left in the hydrogen for some time before heating it, and the author attributes the result to the formation of oxyhydrogen gas in the pores of the platinum, and its explosion when the wire is heated. He considers that the results of the investigation afford further support to his former conclusion that gases are incapable of being statically charged with electricity. G. W. T.

Conductivity of Fused Mixtures of Sodium and Potassium Nitrates. By E. Bouty and L. Poincaré (*Compt. rend.*, **107**, 332—334).—The electric conductivity of fused mixtures of the two salts in widely varying proportions at temperatures between 306·7° and 346·6° agrees closely with that calculated from the known resistances of the constituents. In no case does the difference between the observed and calculated value exceed 0·3 per cent. C. H. B.

Present Condition of the Theory of the Electrolysis of Solutions. By F. Kohlrausch (*Elektrotechnische Zeitschrift*, **8**, 258—265).—This paper contains a valuable summary of the results of researches in electrolysis from the discovery of the electrolysis of water by Nicholson and Carlisle (*Gilbert's Ann.*, **16**, 340) in 1800 down to the present time.

The hypothesis of Grothuss (*Gehlen's Journal*, **8**), propounded eight years later, that electrolytic compounds consist of molecules with opposite electrifications of their own, and that during electrolysis they are separated and travel in opposite directions through the liquid, still forms the essential basis of all inquiries. This was first expressed quantitatively by Faraday (*Exp. Res.*, 7th Ser.) in the statement that the quantity of any constituent separated will be proportional to the quantity of electricity which has traversed the electrolyte, and to the chemical equivalent of the constituent, but will be independent of the compound from which the constituent is taken. This statement, however, is not strictly true of the more complex compounds.

Faraday's law may, from his own researches, together with those of Soret, Buff, Rayleigh, Shaw, and others, be accepted as an exact one with the exception mentioned above; and it follows that each of the constituents into which a molecule is broken up must retain a constant positive or negative charge throughout its electrolytic movements.

About 20 years after the statement of Faraday's law, Hittorff (*Ann. Phys. Chem.*, **89**, 177; **98**, 1; **103**, 1; **106**, 337) showed that while the constituents into which each molecule is divided do travel in opposite directions, they travel with unequal velocities.

The results of Hittorff's researches point to the conclusion that the water of the solution plays no part in the decomposition, and that no appreciable part of the conductivity is due to it. This is only a special case of the more general experimental fact that, with the exception of metals, almost all liquids which are good conductors consist of mixtures of two or more chemical compounds (F. Kohlrausch, (*Ann. Phys. Chem.*, **15**, 271).

The author points out that the investigations of Buff, Poggendorff, Hittorff, von Helmholtz, and others have made it quite impossible to assume that electrolytes conduct like metals, and that electrolysis is a mere secondary effect. This conclusion is in agreement with the fact that electrolytic conductivity is quite independent of transparency to light and radiant heat (F. Kohlrausch, *Ann. Phys. Chim.* [2], **6**, 29; S. Bidwell, *B.A. Report*, 1886, 309).

In spite of this, however, it is found that electrolytes obey Ohm's law, even with such small E.M.Fs. as the millionth of a volt acting through a thickness of a centimetre of liquid, and no effects due to inertia have been observed, even with rapidly alternating currents or currents of short duration (Kohlrausch and Hippoldt, *Ann. Phys. Chem.*, **138**, 370; Cohn, *ibid.* [2], **21**, 667).

The author gives a series of interesting curves representing the conductivity of various substances in solution, the molecular strength of the solution being taken as abscissa, and the corresponding conductivity as ordinate. The curves are all found to be of the same general character, rising more or less rapidly to a maximum as the strength increases, and then diminishing again. The curves show very clearly that similarity of chemical constitution carries with it a similarity in the march of the conductivity. This similarity in the curves is no longer observed if the strengths of the solutions are

given in terms of the percentage composition instead of in terms of the chemical equivalents, which gives an additional proof of the fact that the chemical equivalent is the natural unit of measurement in electrolytic phenomena.

With regard to the influence of temperature, it is found that substances which behave in nearly the same way at any given temperature undergo similar changes in conductivity when the temperature varies. The conductivities of different solutions are found to tend towards equality as the temperature increases (F. Kohlrausch, *Ann. Phys. Chem.* [2], **6**, 191 and 196).

In the case of sulphuric acid, the curve, after reaching a maximum when the solution contains about 30 per cent. of sulphuric acid, falls to a minimum when the proportions of acid and water are such as to form the crystallisable compound $H_2O + H_2SO_4$, confirming the conclusion that the conductivity is less for a chemical compound than for a mixture. It then reaches a second maximum and falls to a minimum again when the solution consists of pure sulphuric acid. If the anhydrous acid is now added the conductivity again increases slightly, and ultimately falls almost to zero. It is instructive to note that the conductivity of the pure sulphuric acid is much less than that of the less stable compound $H_2O + H_2SO_4$.

The curve for sulphuric acid does not coincide with those for nitric and hydrochloric acids, which confirms the theory that the former is a bibasic while the latter are monobasic acids. When, however, the solutions are extremely dilute, the curves agree much more closely, from which it would appear that sulphuric acid when very largely diluted with water tends towards the constitution of a monobasic acid.

There is found to be a distinct connection between the mechanical viscosity of solutions and their conductivities, a series of salts arranged in order of conductivity agreeing in general with the series arranged in order of fluidity. The maximum value of the conductivity also is usually attained the sooner, the greater the viscosity.

A number of instances are known in which substances continue to conduct electrolytically, even when they become solid, and it is found that very little effect is produced on the conductivity of a solution by solidifying it by the addition of gelatin. This, however, is not in any contradiction with the relations between viscosity and conductivity, for viscosity and mechanical rigidity are by no means identical, and, moreover, gelatinous bodies are not homogeneous.

In the case of solutions of electrolytes with monobasic acids, it is found that every constituent has a certain definite speed of its own, whatever be the compound in which it occurs. The author gives the following table of ionic speeds for a molecular concentration 0·1:—

	K.	NH_4.	Na.	Li.	Ag.	H.	Ba.	Mg.	Zn.
+	51	50	33	25	42	272	31	25	24

	Cl.	I.	NO_3.	ClO_3.	$C_2H_3O_2$.	OH.
−	54	54	48	43	27	146

The sum of the speeds of the two constituents of a salt will give its molecular conductivity, that is, the ratio of the conductivity to the

number of electrolytic molecules in unit volume of the solution. The numbers thus obtained in various cases are found to be in close agreement with those determined by direct experiment. As examples of the absolute velocities of the ions, that of hydrogen is found to be in millimetre-seconds 0·3, of potassium or chlorine 0·06, of lithium 0·028 for a slope of potential of 1 volt, and a molecular concentration of 0·1. They would of course be greater in more dilute solutions.

These numbers, of course, are only the mean values of the components of the velocity in the direction of the current, for the electrolytic movements cannot be regarded as being of such a nature that all the constituents should have identical velocities at any given moment.

In connection with the fact that electrolysis depends on differences in the motion of the constituent molecules, it is interesting to note that electrolytes which are good conductors generally diffuse rapidly in water, and the converse also holds, from which it follows that those substances which allow great freedom of motion to the constituent molecules, are in general those in which the entire molecules move most freely. Since electrolytes obey Ohm's law, and since also decomposition begins with any E.M.F., however small, we cannot assume that the molecules are broken up by the E.M.F. Clausius, therefore, assumed (*Ann. Phys. Chem.*, **101**, 338) that in an electrolyte a certain number of the molecules are dissociated, possibly by the action of the water, and that these free molecules are the first which are acted on by the electrical forces, the decomposition then extending by interchanges to the others.

If it is also assumed that the chemical action of an acid or other substance starts, not from the molecules in combination, but from the dissociated molecules, then Clausius's theory gives a simple explanation of the observed relation between conductivity and rapidity of chemical action.

The original paper contains a large number of references to original memoirs, of which a few only are given above. For the others reference must be made to the original paper, or to a translation in *The Electrician*, **21**, 466—467 and 504–507. G. W. T.

Electrolysis with Alternating Currents. By E. Drechsel (*J. pr. Chem.* [2], **38**, 75—77).—The author complains that Manœuvrier and Chappuis (this vol., p. 1005) have not acknowledged his work on this subject. The spontaneous detonations which occur when water is electrolysed by alternating currents are not due to the incandescence of the electrodes, nor need the latter come in contact with a vessel of the mixed gases, the detonations occurring even when the electrodes are fully immersed in the liquid; the author attributes them to sparking. A. G. B.

Production of Ozone by the Electric Discharge. By Bichat and Guntz (*Compt. rend.*, **107**, 334—336).—The ozoniser employed consisted of a platinum wire, 0·1 mm. in diameter, stretched along the axis of a cylinder of the same metal through which passed a slow

current of oxygen at constant pressure. The wire was connected with a Holtz machine and an absolute electrometer, and the cylinder was connected with the earth and a galvanometer. By means of a Mascart's "Trop-plein" a constant discharge was obtained.

For the same intensity of discharge, the negative effluve produces 10 times as much ozone as the positive effluve. If the discharge takes place between a platinum point and a disc of the same metal, the difference is less marked, but is still very distinct. This result is explained if it is assumed that the production of ozone is not a result of electrification, but of a rise of temperature, since the negative discharge is hotter than the positive.

For the same velocity of gaseous current, the quantity of ozone produced increases with the intensity of the discharge and the difference of potential, but there is no simple quantitative relation. At first the quantity of ozone formed varies as the square of the potential; but this only holds good for potentials up to 20 C.G.S. Faraday's law does not apply.

In the ozone apparatus devised by Berthelot, it is seen when the discharge takes place in the dark that the oxygen is illuminated by an infinite number of minute sparks commonly termed a "rain of fire," this being more brilliant the lower the resistance. The quantity of ozone also varies with the resistance, and is lower the less brilliant, or, in other words, the cooler, the discharge. In the same apparatus a discharge which produces no sparks produces no ozone. It is evident from these observations that the formation of ozone cannot be due to a dielectric polarisation or to a state of strain resulting from it. In Berthelot's apparatus, the quantity of ozone formed has no relation to the difference of potential between the armatures. The capacity is constant, and therefore Faraday's law is applicable.

The production of ozone is due to a more or less considerable elevation of the temperature of the oxygen resulting from the passage of the electric discharge. The oxygen, in fact, is under conditions similar to those in a Deville's hot-and-cold tube.

With an ozoniser consisting of a metallic point and disc, only 0·4 per cent. of the energy of the discharge is utilised in the production of ozone. With Berthelot's apparatus, on the other hand, as much as 89 per cent. of the energy may be utilised. C. H. B.

Electrical Dialysis. By H. N. Warren (*Chem. News*, **58**, 76).—The author has devised an arrangement for the purpose of converting a substance "into an allied form" by the aid of electrolytical nascent hydrogen, and at the same time of dialysing the product. A tube closed at the bottom by a parchment diaphragm serves as the dialyser, and is fitted with a cork carrying a tube with some mercury in it, and connected to a pendant platinum plate by a wire of the same metal, fused through the lower end. This dialyser contains the substance to be acted on, and is suspended in an outer vessel, containing—1, an aqueous solution of the reagent which is to combine with the substance; 2, a porous pot, in which is placed some solid ammonium chloride, a zinc rod, and some water. The liquids in all three vessels are adjusted to the same level, the platinum plate in

the dialyser is made the negative electrode, and the zinc rod the positive pole of a galvanic circuit. With copper sulphate and sodium chloride in the dialyser, and dilute sulphuric acid in the outer vessel, copper is deposited on the platinum and sodium sulphate dialyses. Potassium dichromate gives rise to a deposit of chromic hydroxide and a dialysate of potassium sulphate. With ricinoleic acid in the dialyser and sodium carbonate outside, sodium stearate forms. Many other substances have been treated. D. A. L.

The Specific Heats of some Metals from the Ordinary Temperature up to 300°. By A. NACCARI (*Gazzetta*, **18**, 13—31). The author criticises the methods employed by those physicists who have made determinations of the specific heats of metals at high temperatures, and points out the various sources of error in them, condemning especially the methods of Bède and Byström, whose results he considers to be of very little value.

In the apparatus employed by the author, the metal, whose specific heat had to be determined, was heated in a metal basket suspended in an air-bath with double walls, the temperature of which was kept constant by passing the vapour of a liquid through the annular space between these walls. It was so arranged that the metal and the basket could be dropped direct from the air-bath into the calorimeter; this latter was made of brass, and fitted with a stirring apparatus and a delicate thermometer graduated to $\frac{1}{50}$th of a degree. The thermometers employed were carefully compared with the air thermometer, and the 100° point verified from time to time; further, by choosing the thermometer whose scale was best adapted for the temperature in the calorimeter during any given experiment, the correction for the column of mercury not immersed in the liquid could be reduced to a minimum. One of the chief causes of error in methods formerly employed was the use of water as the liquid for the calorimeter; when a metal at a high temperature is plunged into this, steam is produced, and the result is necessarily inaccurate. This inconvenience is obviated by employing high-boiling petroleum, 330—380°, which was carefully rectified, so as to obtain a specimen tolerably homogeneous. Its specific heat was determined from time to time by means of zinc, but it varied very little in the course of a series of experiments.

In the paper, the results of the experiments are given in detail, also a formula of the form—

$$q = A(t - T) + B.10^{-6}(t - T), \quad . \quad . \quad . \quad . \quad (1)$$

expressing the amount of heat required to heat 1 gram of the metal from a low temperature T (15—21°) to a higher temperature t, A and B being constants which vary with each metal. The true specific heat γ being given in another form of the formula—

$$\gamma = dq/dt = A + C.10^{-6}(t - T). \quad . \quad . \quad . \quad . \quad (2)$$

The following are the constants A and C for the various metals examined, and in columns three and four are given the specific heats

at the temperatures 50° and 300° respectively. In the original, they are given for every 50° between these limits.

	A.	C.	γ at 50°.	γ at 300°.
Cadmium	0·055107	23·78	0·0551	0·0617
Zinc	0·0915	44·4	0·0929	0·1040
Iron*	0·10912	58·064	0·1113	0·1376
Silver	0·054984	21·412	0·05556	0·06091
Copper	0·092455	21·258	0·09316	0·09846
Nickel	0·10569	94·60	0·1090	0·1327
Antimony	0·048896	16·718	0·04947	0·05366
Lead	0·02993	13·5846	0·03040	0·03380
Aluminium	0·21235	95·07	0·2164	0·2401

The following are the coefficients of the above formula reduced to—

$$\gamma = a(1 + bt),$$

the metals being arranged according to b.

	a.	$b \cdot 10^6$.
Copper	0·09205	230·8
Antimony	0·04864	343·7
Silver	0·05449	392·9
Cadmium	0·05461	433·4
Aluminium	0·2116	449·3
Lead	0·02973	456·9
Zinc	0·09070	489·5
Nickel	0·10427	907·0
Iron	0·10442	1029·1

C. E. G.

Attempt to Eliminate the Influence of the Change in the Volume of the Vessel when Measuring the Expansion of Liquids. By J. J. Boguski (*Zeit. physikal. Chem.*, **2**, 482—487).—The apparatus is a modification of that used for measuring the compressibility of liquids (this vol., p. 1019), the principle being the measurement of the volume of the liquid in expansion, and in its normal condition in two separate vessels. A determination of the coefficient of expansion of amyl alcohol between 0° and 100° gave the value 0·00107732. H. C.

Thermal Conductivity of Mercury above 100°. By A. Berget (*Compt. rend.*, **107**, 171—172).—Experiments between 100° and 320° show that the value of the coefficient K diminishes as the temperature rises, the variation being 0·00045 for one degree.

Incidentally the author verified the law that in two walls of the

* In the case of iron, it is necessary to add a third term to equation (2), namely, $0{\cdot}146574 \cdot 10^{-6}(t - T)^2$.

same nature but of different diameters, the times required to establish stationary temperatures are to one another as the squares of the diameters of the walls. C. H. B.

Alloys of Potassium and Sodium. By A. JOANNIS (*Ann. Chim. Phys.*, **12**, 358—384).—The author determined the quantity of heat which is generated, (1) when alloys of potassium and sodium are dissolved in water, (2) when the separate metals are dissolved in water, and (3) the latent heat of fusion of the separate metals. The following are the results (in Calories) as the average of several experiments in each case:—

(1.) Na_2K ... 44·50, NaK ... 44·06, NaK_2 ... 43·81, NaK_3 ... 44·40.
(2.) K ..... 45·39, Na .... 42·59
(3.) K ..... 0·61, Na .... 0·73

These results show that in the formation of the alloy Na_2K, there is absorption of heat, whereas in the formation of NaK there is a slight development of heat (1·20 Cal.).

The only alloy which has a definite composition is NaK_2, the formation of which is accompanied by the development of 3·89 Cal. The development of heat (3·72 Cal.) attending the formation of NaK_3 is less than in the production of NaK_2, showing that the former is probably a solution of potassium in the alloy NaK_2. F. S. K.

Sodium Glycol-oxide. By DE FORCRAND (*Compt. rend.*, **107**, 343—345).—23 grams of sodium is not completely dissolved by 62 grams of glycol even when heated for some time, and afterwards evaporated at 200° in a current of hydrogen. About one-seventh of the sodium remains undissolved. No better result is obtained by adding glycol to an alcoholic solution of sodium ethoxide.

If the compound $C_2H_5NaO,3C_2H_6O$ is mixed with one equivalent of glycol, heated in an oil-bath, and finally evaporated at 105—110° in a current of pure and dry hydrogen, 4 mols. of ethyl alcohol are given off, and a white residue is obtained which is not quite free from glycol. The *monosodium glycol* thus obtained does not alter in dry air, but is very deliquescent and very soluble in water. Heat of dissolution at 20° = +6·01 Cal.; heat of dissolution of glycol at the same temperature = +1·65 Cal. Treatment of the glycol solution with sodium hydroxide and of the sodium-derivative with sulphuric acid gave the following results:—

$C_2H_6O_2$ liq. + $\frac{1}{2}Na_2O$ solid = $C_2H_5NaO_2$ solid + $\frac{1}{2}H_2O$ solid	develops	+24·77 Cal.
$C_2H_6O_2$ liq. + NaHO solid = $C_2H_5NaO_2$ solid + H_2O solid	„	+7·76 „
$C_2H_6O_2$ liq. + Na solid = $C_2H_5NaO_2$ solid + H, gas	„	+39·65 „

In its thermochemical behaviour, glycol is intermediate between the monhydric and trihydric alcohols. C. H. B.

Potassium and Sodium Malonates. By G. Massol (*Compt. rend.*, **107**, 393—395).—*Sodium malonates.*—

$C_3H_4O_4$ solid + $\frac{1}{2}Na_2O$ solid	develops	+13·05 Cal.
$C_3H_3NaO_4$ solid + $\frac{1}{2}Na_2O$ solid	„	+13·60 „

The heat of dissolution of sodium hydrogen malonate = −6·616 Cal.

$C_3H_4O_4$ solid + NaHO solid = $C_3H_3NaO_4$ solid + H_2O solid	develops	+25·86 Cal.

Normal sodium malonate seems to form a hydrate containing 1 mol. H_2O with a heat of dissolution +0·13 Cal., and another containing $\frac{1}{2}$ mol. H_2O, with a heat of dissolution +1·5 Cal. Heat of dissolution of the anhydrous salt = +3·08 Cal.

$C_3H_4O_4$ solid + 2NaHO solid = $C_3H_2Na_2O_4$ solid + $2H_2O$ solid	develops	+41·49 Cal.
$C_3H_2Na_2O_4$ solid + $\frac{1}{2}H_2O$ liq. = $C_3H_2Na_2O_4,\frac{1}{2}H_2O$ solid	„	+1·58 „
$C_3H_2Na_2O_4$ sol. + H_2O liq. = $C_3H_2Na_2O_4,H_2O$ solid	„	+3·03 „

Potassium Malonates.—

$C_3H_4O_4$ solid + $\frac{1}{2}K_2O$ solid.....	develops	+13·36 Cal.
$C_3H_3KO_4$ solid + $\frac{1}{2}K_2O$ solid.....	„	+13·94 „

The acid salt crystallises with $\frac{1}{2}$ mol. H_2O.

$C_3H_4O_4$ sol. + KHO sol. = $C_3H_3KO_4,\frac{1}{2}H_2O$ solid + $\frac{1}{2}H_2O$ solid	develops	+31·66 Cal.

Normal potassium malonate crystallises with 2 mols. H_2O; heat of dissolution = −5·0 Cal. Heat of dissolution of the anhydrous salt = +2·10 Cal.

$C_3H_4O_4$ solid + 2KHO solid = $C_3H_2K_2O_4$ solid + $2H_2O$ solid.....	develops	+48·56 Cal.
$C_3H_2K_2O_4$ solid + $2H_2O$ liquid = $C_3H_2K_2O_4,2H_2O$ solid	„	+7·10 „

In every case the thermal disturbance is less than for the corresponding reaction with oxalic acid. C. H. B.

Heat of Formation of Toluidines, Benzylamine, and Methylaniline. By P. Petit (*Compt. rend.*, **107**, 266—269).—The heats of combustion were determined by means of the calorimetric bomb, and the results are given in the following table:—

	Heat of combustion.			
		Per gram-molecule.		
	per gram.	Constant pressure.	Constant volume.	Heat of formation.
Orthotoluidine..	9·007	964·7	963·75	3·8
Metatoluidine ..	9·015	965·6	964·6	2·9
Paratoluidine...	8·952	958·8	957·86	9·7
Benzylamine....	9·043	968·6	967·6	0·1
Methylaniline...	9·094	974·0	973·06	−5·5

The heats of formation are calculated for the diamond and gaseous nitrogen and hydrogen. If the heat of fusion of paratoluidine is deducted, it will be seen that the heats of formation of the three toluidines do not differ by quantities sensibly greater than the error of experiment. The formation of orthotoluidine from orthocresol and ammonia with elimination of water would develop about +18 Cal.

The formation of benzylamine from benzyl alcohol and ammonia with elimination of water would develop +12·2 Cal.

It is evident that methylaniline is not analogous to the other compounds. Its formation from phenol, methyl alcohol, and ammonia, with elimination of 2 mols. H_2O, would develop +24·8 Cal., and its formation from aniline and methyl alcohol, with elimination of H_2O, would develop +15 Cal. These results explain the transformation of salts of methylaniline into salts of paratoluidine. The change of the one base into the other would develop about +15 Cal., and the union of acids with paratoluidine develops more heat than the union of the same acids with methylaniline. C. H. B.

Heat of Neutralisation of Malonic Acid. By G. Massol (*Compt. rend.*, **107**, 257—260).—Heat of dissolution (104 grams in 4 litres) at 20°, −4·49 Cal. Gal and Werner found −4·573 Cal. at 10°.

The heats of neutralisation in dilute solution are as follows:—

	NaHO.	KHO.	AmHO.	$Ba(HO)_2$.	$Sr(OH)_2$.	$Ca(OH)_2$.
Acid salt....	13·05	13·36	12·14	13·39	13·485	13·49
Normal salt..	13·60	13·94	12·90	16·64*	13·415	13·60
Total	26·65	27·30	25·04	30·13	26·90	27·09

It will be seen that in all cases the values are higher than those for oxalic acid, but lower than those for succinic acid.

C. H. B.

Influence of the Shape of the Bulb in Vapour-density Determinations. By H. Biltz (*Ber.*, **21**, 2772—2776; compare Biltz, this vol., p. 1027; *Ber.*, **21**, 2016).—The following experiments were made to determine the influence of the shape of the bulb in

* Partially precipitated.

vapour-density determinations by the displacement method in cases where the substance is not completely converted into the gaseous state under the conditions of the experiment.

The vapour-density of sulphur at 518°, employing a bulb 30 cm. long and 3·3 cm. in diameter, was found to be 5·0 as the mean of many closely agreeing experiments, whereas with a bulb 26 cm. long and 7·5 cm. in diameter, the average value obtained was 4·5. Experiments with methylene bromide at 100° gave similar results; in an apparatus the bulb of which was 27 cm. long and 2·2 cm. in diameter, the vapour-density was found to be 6·4 on the average, and with a bulb 26 cm. long and 7·5 cm. in diameter, 6·1 as the average of six concordant experiments. F. S. K.

Method of Estimating the Molecular Weight of Volatile Chlorides. By H. Biltz (*Ber.*, **21**, 2766—2772).—The author determined the vapour-density of indium chloride and ferric chloride by heating a known weight of the metal in an atmosphere of dry chlorine at a suitable temperature, and measuring the change in volume which took place. The apparatus, a modification of V. Meyer's apparatus for the determination of vapour-density, is fully described with the aid of a diagram.

The author found that at a bright red heat the vapour-density of indium chloride is 9·565, corresponding with the molecular formula $InCl_3$ (compare V. Meyer, *Ber.*, **12**, 611; Nilson and Pettersson, Trans., 1888, 816). The specific gravity of ferric chloride at 518° was found to be 9·388 as the average of three experiments. (Compare *loc. cit.*; also Grünewald and Meyer, this vol., p. 422.) F. S. K.

Hydrates of Methane and Ethylene. By Villard (*Compt. rend.*, **107**, 395—397).—*Methane Hydrate.*—The pure gas was compressed with varying quantities of water in Cailletet's apparatus.

Temperature	0°	+1·1°	5·5°	8·5°	9·9°	10·8°	14·3°
Tension in atmos. above normal..	26·5	30	47	63·5	75	83	123·5

Temperature	16·1°	17·3°	19·3°	20·3°
Tension in atmos. above normal.....	152	178·5	232	265

The tension is independent of the proportion of water present. The critical temperature of the hydrate is 21·5, and it slowly decomposes at this temperature under a pressure of 300 atmos. According to Dewar the critical temperature for the gas is −99·5°.

Ethylene Hydrate.

Temperature	0°	+3°	5·5°	8°	11°	13·4°	14·8°	16·6°	17·2°
Tension in atmos. above normal..	6·5	8·5	11	14	21	28·5	34·5	45	59

At −18·7 the hydrate slowly decomposes under any pressure.

According to Dewar the critical point of ethylene is +10·1; a redetermination by the author gave +10° as the critical temperature, and 51 atmos. as the critical pressure. The critical point of ethylene itself is the same in the presence of moisture as when dry.

C. H. B.

Freezing Points of Dilute Aqueous Solutions. By F. M. RAOULT (*Zeit. physikal. Chem.*, **2**, 488—490).—The author gives the details of his experiments with those substances, for which the values of i calculated from the freezing points of the aqueous solutions differed most from those calculated by Arrhenius from the conductivity (this vol., p. 896). H. C.

Freezing Points of Dilute Aqueous Solutions. By S. ARRHENIUS (*Zeit. physikal. Chem.*, **2**, 491—505).—The author has redetermined the freezing points of those aqueous solutions, which, using Raoult's determinations, gave numbers for i which were not in sufficient agreement with those calculated from the conductivity (preceding Abstract). The result of this work has been to bring the two into closer agreement. H. C.

Apparent Manifestation of Chemical as Mechanical Attraction. By S. KALISCHER (*Zeit. physikal. Chem.*, **2**, 531; see this vol., p. 1009).—It is pointed out that the results obtained by Langley are in accordance with those of Faraday, who, with a cell consisting of zinc and platinum in sulphuric acid, observed that on completing the circuit a decrease in the current first occurred, and that it subsequently attained its proper value. H. C.

The Dead Space in Chemical Reactions. By O. LIEBREICH (*Berlin. Akad. Ber.*, **43**, 959—962).—It has hitherto been assumed that chemical reaction takes place uniformly in all parts of a thoroughly mixed liquid, provided that there are no currents, and that the temperature is uniform. In this paper experiments are described which show that there may be a space in mixed solutions in which no reaction takes place. This is best shown by mixing aqueous chloral hydrate and aqueous sodium carbonate. When the solutions are suitably diluted, the chloroform which is produced separates as a fine cloud, and the time required for the reaction to commence may be varied from 1 to 25 minutes. When a test-tube is used a space from 1 to 3 mm. below the meniscus remains clear, and the lower portion of the liquid in which the reaction takes place is sharply divided from the upper by a surface curved in a direction opposite to the meniscus; this is distinct even after 24 hours. This dead space was also observed in vessels of various forms. In the case of a very small drop of the mixture, the constituents of which had been previously boiled, which was contained in a capillary tube, no reaction took place at all. When closed vessels quite full are used the reaction takes place uniformly throughout the whole, but when a full tube is closed with an animal membrane, it is possible to show the dead space. If a little of the still clear liquid is removed by means of a capillary tube and warmed, the reaction at once takes place.

Similar experiments with sulphurous and iodic acids are described. The sulphurous acid was of such a strength that 5 c.c. of the aqueous solution just decolorises 2 c.c. of a 1 per cent. permanganate solution.

The following conclusions are drawn:—(1). In liquids the space of the chemical reaction is limited by a zone in which no reaction takes place, this zone being that portion of the liquid which is in contact with the air, or separated from the air by a thin membrane. (2.) That the reactions take place more slowly in narrow than in wide tubes. (3.) A capillary space is capable of preventing a reaction from taking place. N. H. M.

Compression of the Moist Powder of Solid Substances and the Formation of Rocks. By W. Spring (*Zeit. physikal. Chem.*, **2**, 532—535).—The author shows that the conglomeration of the moist powder of different chemical substances under pressure up to 6000 atmospheres is in some cases assisted, and in others retarded, by the presence of water, the quantity of the latter used in each case being 2 drops per c.c. With all metals, the action of the water is a retarding one. With soluble substances, the conglomeration is assisted for those which give a solution the volume of which is less than, and retarded for those which give a solution the volume of which is greater than the sum of the volumes of the solvent and the soluble substance. Substances belonging to the former of the above two classes have their solubility in water increased by pressure, the solubility of those belonging to the latter being decreased. Insoluble substances do not show such marked differences in the wet and dry state, but in some cases the presence of water appears to assist the conglomeration. H. C.

Chemical Action between Substances in the Solid State. By W. Spring (*Zeit. physikal. Chem.*, **2**, 536—538).—It is found that reactions conditioned by temperature and pressure take place between various substances in the solid state, and it appears that the particles of solid matter possess a power of diffusion similar to that of gases and liquids, although of course in much smaller degree. Thus if dry barium sulphate and sodium carbonate are mixed and compressed, a reaction sets in, which gradually spreads throughout the whole mass, and can be greatly accelerated by heat. Finely divided copper mixed with dry mercuric chloride was left to itself in a sealed tube, being only shaken from time to time in order to produce fresh contact. In four years' time the reaction was complete, and had resulted in the formation of cuprous and mercurous chlorides. In the same way dry potassium nitrate acts on dry sodium acetate, when the two are mixed in the form of powder, potassium acetate being formed. The author proposes to further investigate these changes. H. C.

Determination of the Velocity of Etherification by Means of Electrical Conductivity. By Negreano (*Compt. rend.*, **107**, 173—176).—An extension of the method previously employed in the case of mixtures of acetic acid and ethyl alcohol in equal molecular proportions to mixtures containing an excess of one or the other. (Compare this vol., p. 1025.) C. H. B.

The Energy of Compounds and the Oxides of Potassium and Lithium. By N. Beketoff (*Chem. Centr.*, 1888, 994, from *Bull. Acad. St. Pétersbourg*, **12**, 743).—The author suggests the hypothesis that a compound of two elementary substances is stronger the nearer the proportion between the weights of the elements approaches to unity. As an example in support of this, potassium oxide is reduced by hydrogen at a low heat, whereas lithium oxide is not reduced by hydrogen or carbonic oxide. The author calculates also that the heat of oxidation of rubidium and cæsium is smaller than that of potassium, and that their oxides must be reducible by hydrogen.

J. W. L.

Aspirator with Constant Flow. By O. Binder (*Zeit. anal. Chem.*, **27**, 465—466.)—In aspirators of the usual form, the head of water is constantly diminishing, and the rate of aspiration is therefore not constant. But by carrying the tube conveying the gas down to the bottom of the aspirator, the effective column of water remains constant. If it be desired to diminish the action of the water on the aspirated gas, the tube may be surrounded by a larger one, open at the top, and fitted at the bottom with a narrow recurved tube; the bubbles of gas then come in contact only with the water in the wide tube. The water escape-tube should pass to the bottom of a Woulff's bottle containing a little mercury. This forms a valve, and prevents regurgitation.

M. J. S.

Safety Retort for Preparing Gases. By N. v. Klobukoff (*Zeit. anal. Chem.*, **27**, 467—469).—The retort consists of three parts: a cast-iron cylindrical pot, with a gutter about 2 cm. deep round its edge; a cast-iron convex cover, with a vertical rim fitting into the gutter; a wrought-iron tube screwed into the cover. The gutter is filled with a pulp consisting of 100 parts of coarse sand and 50 or 60 parts of plaster of Paris. This mixture when dry is but little penetrable by gases even under moderate pressure (30 cm. of water), but yields easily in the event of any high pressure in the retort, allowing the cover to lift, and thus preventing explosion.

M. J. S.

Lecture Apparatus for Showing Combustion of Air in Coal-gas. By G. Craig (*Chem. News*, **58**, 55).—The apparatus consists of an inverted flask with a side-tube in the neck and an orifice at the bottom lined with asbestos; the mouth is fitted with a cork soaked in vaseline through which passes a glass tube. The flask is filled with coal-gas through the side-tube which is ignited at the orifice, the tube is pushed up through the cork, and the air will soon be seen burning from the tube with its usual feebly luminous flame in the interior of the flask.

D. A. L.

Combustion of Oxygen in Ammonia and of Hydrogen in Nitric Acid. By W. R. Hodgkinson and F. K. Lowndes (*Chem. News*, **58**, 27).—For the combustion of oxygen in ammonia, a long platinum jet about 1 mm. bore, and 8 or 9 cm. long is introduced into a flask containing 0·880 ammonia to within a cm. of the surface of the liquid, the escaping gaseous mixture is then ignited. The

oxygen burns at the jet which becomes white hot, and when immersed in the ammonia the oxygen continues to burn, generally melting the platinum tube. By regulating the pressure of oxygen, fumes of ammonium nitrite and nitrate are obtained.

When a hydrogen flame is introduced in a similar manner into a flask of nitric acid to within 2 cm. of the surface of the acid, and the pressure is adjusted so that the flame just touches the surface, the jet soon becomes white hot, and a cone of coloured flame is seen to extend from the neck of the flask to the surface of the liquid, while fumes of ammonium nitrite and nitrate escape. If the white hot point is immersed in the acid, nitrous fumes also escape, and the hydrogen continues to burn until the acid becomes considerably diluted.

D. A. L.

Inorganic Chemistry.

Reducing Action of Hydrogen in Presence of Platinum. By S. Cooke (*Chem. News*, **58**, 103—106).—In these experiments, various solutions were exposed with and without platinum to the action of hydrogen prepared from pure dilute sulphuric acid, either electrolytically, or by the action of zinc. In the platinum experiments, strips of the metal as long as the tubes, and nearly as wide, are placed in tubes 300 to 400 mm. long, and about 15 mm. wide, and are held in position by a piece of platinum wire passing through the closed end of the tube. The platinum strip is coated, electrolytically, with platinum black, the tube is then filled with the solution under trial, which is subsequently displaced by hydrogen, and in this condition the tube remains in the solution. In this manner, nitric acid varying in strength from sp. gr. 1·420 to $\frac{1}{64}$ normal solution was submitted to trial; the action in dilute acids, and also in the experiments with nitrates referred to below, were aided by heating. With the strong acids, red fumes soon appear, and hydrogen is absorbed, nitrous acid being produced; with weak and dilute acids, the same action occurs in a less marked degree and at a slower rate, and ammonia is produced as well as nitrous acid. In a similar manner, potassium, sodium, barium, calcium, cobalt, nickel, lead, copper, and silver nitrates, also potassium permanganate, dichromate, and ferricyanide, and ferric chloride and sulphate, platinum chloride, mercuric chloride, and sulphuric and sulphurous acids were submitted to experiment, and likewise suffered reduction. Moreover, in the presence of the coated platinum, chlorine, bromine, and iodine combine with hydrogen in the dark; solutions of chlorine and bromine in water and iodine in potassium iodide were employed in these experiments, since with gaseous chlorine and bromine the coating frequently comes off. Hypochlorous acid and potassium chlorate are also reduced, but potassium perchlorate is not acted on. The only instances of the action of hydrogen alone on the above substances were observed in the experiments with silver nitrate,

platinum chloride, and potassium permanganate and ferricyanide, and in all cases the action was considerably slower than when in the presence of the coated platinum; it is noteworthy that the author observed no reduction when concentrated sulphuric acid or solutions of copper nitrate were exposed to hydrogen alone. Similar experiments were made with hydrogen and nitric oxide either over water or with the moist mixture over mercury. One volume of hydrogen to two volumes of nitric oxide reacted regularly in the manner represented by the equation $H_2 + 2NO = N_2O = H_2O$; equal volumes of the two gases gave a contraction equal to half the original volume, the residue being nitrogen, whereas with a large excess of hydrogen, 600 c.c. to 150 c.c. of nitric oxide, the contraction amounted to 300 c.c., the residual gas consisting almost entirely of hydrogen with a little nitrogen. All nitric oxide had disappeared, and the liquid was strongly alkaline, and contained hydroxylamine and ammonia, but no nitrous oxide. With nitric oxide in smaller proportion, the action appears pretty much the same but is slower. Nitric oxide is also reduced in a similar manner, but to a less extent, when zinc coated with copper is used instead of platinum; but with platinum, nitric oxide and carbonic oxide yield nitrous oxide and nitrogen in proportions varying with the amount of carbonic oxide used. Moreover, nitric oxide is reduced by ammonia and nitrous oxide by hydrogen, ammonia, and carbonic oxide. The platinum is more active when freshly prepared. D. A. L.

Removal of Iodate from Iodide of Potassium. By H. N. Morse and W. M. Burton (*Amer. Chem. J.*, **10**, 321—322).—The solution of the iodide is boiled some little time with zinc amalgam, then filtered; the filtrate is free from zinc and mercury. The amalgam is prepared by agitating zinc-dust with mercury in presence of tartaric acid, and subsequently washing with water. Iodate of potassium is reduced by this reagent more quickly than the bromate or chlorate. H. B.

Valency of Boron. By R. Lorenz (*Annalen*, **247**, 226—251).—As boron trioxide is exceedingly hygroscopic, an intimate mixture of the oxide with charcoal can only be obtained free from moisture by fusing boric acid and charcoal in a platinum crucible. When the mixture is in a state of quiet fusion, it is cooled and transferred to a tube of refractory glass, and heated in a current of dry carbonic anhydride to expel traces of moisture. When chlorine is passed over the mixture, boron trichloride and oxychloride and benzene hexachloride are produced. The oxychloride remains as a white, fibrous mass when the crude boron chloride is distilled. It has the composition $B_8O_{11}Cl_2$, and not $BOCl_3$ as stated by Councler (*J. pr. Chem.* [2], **18**, 371). According to Gustavson (*Zeit. Chem.*, **6**, 521), the oxychloride BOCl is formed when equivalent quantities of boron trichloride and trioxide are heated at 150° in sealed tubes. The author, however, finds that the product has the composition $2BCl_3 + 7B_2O_3$.

Boron prepared by the reduction of boric acid with sodium or

potassium is invariably mixed with a small quantity of a solid hydride of boron.

At a red heat, amorphous boron decomposes carbonic anhydride, carbon is set free, and boron trioxide is formed. As the chief argument in favour of the pentavalency of boron was the supposed existence of the oxychloride $BOCl_3$, the element may now be regarded as trivalent. W. C. W.

Boric Acid. By P. Georgievič (*J. pr. Chem.* [2], **38**, 118—120).—In this paper the author pleads for a position for boron in the aluminium group in a periodic classification of the elements, on account of (1) its oxide acting like aluminium oxide, both as a feeble base, in such compounds as BPO_4 and $B_2(SO_4)_3 + H_2O$, and as a feeble acid; (2) the similarity of the alkyl compounds of boron and aluminium; (3) the isomorphism of euclase and datolite.

The feeble acid character of boric acid is proved by the facts that (1) a solution of an alkaline carbonate is not decomposed by it; (2) a solution of borax is completely decomposed by a current of carbonic anhydride, and barium borate is dissolved when suspended in water through which hydrogen sulphide is passed; (3) boric acid will not liberate iodine from a mixture of potassium iodide and iodate or nitrite: (4) the red colour of ferric acetate and the violet colour of ferric chloride in solution of phenol are not bleached by boric acid; (5) boric acid is liberated from borax by the action of iodine, sodium iodide and iodate being formed.

Ostwald's avidity equivalent of 3·01 for boric acid is too high.

A. G. B.

Atomic Weight of Zinc as determined by the Composition of the Oxide. By H. N. Morse and W. M. Burton (*Amer. Chem. J.*, **10**, 311—321).—Pure zinc of commerce was distilled in a vacuum, using a glass tube divided into three by constrictions: from the first division the metal was distilled into the second and third divisions; when about one-fourth still remained in No. 1 division and about one-tenth had passed into No. 3 division, the operation was stopped and the process repeated four times on the contents of No. 2 division. The purified metal was found spectroscopically to be free from lead, cadmium, tin and indium. The nitric acid used for solution of the metal was distilled from glass and condensed on the outside of a platinum basin cooled by ice; thus avoiding contamination from gold solder. The solution, evaporation, and ignition were effected in porcelain crucibles, and to prevent the lids, &c., from sticking together by fusion of the glaze, this last was when necessary removed by hydrofluoric acid. The crucible was balanced by an exactly similar one which was treated with acid and ignited just as the one in actual use. After drying and heating in an air-bath, the crucibles were heated in a muffle for three hours to above the fusing point of steel. Fifteen determinations of the atomic weight varied from 65·091 to 65·119, with a mean of 65·106 (O = 15·96). Marignac has stated that zinc oxide dissociates at high temperatures, and that even at this temperature it still retains oxides of nitrogen. The first

statement the authors have already shown not to be true (this vol., p. 652), and the latter statement has not held in the work here described. H. B.

Note.—The authors carefully describe the following precautions to be taken for testing for nitrogen oxides in the weighed zinc oxide: viz., removal of chlorine from the hydrochloric acid used for the solution of the sample by agitation with mercury, removal of traces of iodate from the iodide of potassium, and they remove the dissolved oxygen from the reagents by boiling, but they effect the test itself at a boiling temperature, at which the iodide of starch for which they are seeking could not possibly be formed. H. B.

Phosphorescent Hexagonal Blende. By A. Verneuil (*Compt. rend.*, **107**, 101—104).—The author has shown (this vol., p. 791) that zinc sulphide sublimed in a slow current of hydrogen is not phosphorescent. Natural or artificial blende distilled at an orange-red heat in pure and dry hydrogen sulphide, yields a product which is not phosphorescent. At a bright white heat, the blende sublimes somewhat rapidly even in a slow current of gas, but in this case also the sublimate is not phosphorescent. The gas which escapes from the apparatus under these conditions is a mixture of hydrogen and hydrogen sulphide containing 71 to 75 per cent. of the latter. If blende is sublimed in an atmosphere consisting of 78 per cent. of hydrogen and 22 per cent. of hydrogen sulphide, the sublimate is only slightly phosphorescent. With only 12 per cent. of hydrogen sulphide, the phosphorescence is brighter. In neither case is there a deposit of sulphur, and analysis fails to detect any difference in composition between the phosphorescent and non-phosphorescent varieties.

When non-phosphorescent blende is distilled at an orange-red heat in an atmosphere of nitrogen, a minute trace of hydrogen sulphide escapes. If the same blende is heated at 400° in a vacuum, a small quantity of the same gas is evolved, and hence it would seem to be occluded in the blende. The sublimate obtained in nitrogen has a feeble white phosphorescence even if sublimation is effected at a bright white heat. Blende mixed with 5 per cent. of zinc oxide or 1 or 2 per cent. of carbon from sugar, and sublimed, yields a sublimate which shows a brilliant phosphorescence. The same result is obtained in a neutral atmosphere if the sublimate is cooled rapidly in order to prevent the re-combination of any products of dissociation. From these results it follows that wurtzite formed by sublimation becomes very phosphorescent only when the original blende has been in contact at a high temperature with some desulphurising agent. The phosphorescence is brighter the lower the temperature at which the wurtzite is formed. C. H. B.

Observations on Sabatier's Papers on Hydrochlorides of Cupric and Cobalt Chlorides. By Engel (*Compt. rend.*, **107**, 178—179).—The author points out that Sabatier's and his own results are not contradictory, but prove the existence of two hydrochlorides of cupric chloride, just as in the case of zinc chloride. He claims

priority in the discovery of the hydrochloride of cobalt chloride. (Compare this vol., pp. 558, 1036, 1037.) C. H. B.

Basic Cupric Chromate. By L. Balbiano (*Chem. Centr.*, 1888, 1024, from *Rend. Acad. Lincei* [4], **4**, 597—600).—A solution of cupric sulphate is not completely precipitated by neutral ammonium chromate, the complete precipitation only being effected by the addition of ammonia. Both the precipitate first produced by the ammonium chromate and that by the later addition of the ammonia have the same composition, $Cu_3CrO_6 + 2H_2O$.

By evaporating the aqueous solution on the water-bath, an amorphous mass is formed. Alcohol precipitates from the aqueous solution a dirty green precipitate, and is at the same time oxidised to acetic acid. J. W. L.

Action of Petroleum on Lead. By W. Fox (*Chem. News*, **58**, 39).—The lead lining of tanks in which petroleum is stored soon becomes corroded, and a brownish powder forms consisting of lead hydroxide, carbonate, and a little valerate and some organic matter. When lead is heated in a sealed tube with petroleum free from acid, no gas is evolved and lead oxide alone is formed; if a little valeric acid is added to the petroleum and oxygen is excluded from the tube, then lead hydroxide and carbonate form; heated in a similar manner with valeric acid in the presence of oxygen, the lead forms hydroxide and carbonate and oxygen is absorbed. The author therefore thinks that the formation of lead carbonate in the petroleum tanks is due to the action of an oxidising agent, and a small quantity of valeric acid present in the petroleum. D. A. L.

Yttrium Compounds. By A. Dubois (*Compt. rend.*, **107**, 99—101 and 243—245).—3 parts of pure yttria from the oxalate, 1 part of amorphous silica, and 30 parts of calcium chloride were heated together for two hours in a wind furnace and the cooled product was treated with water. In this way a silicate, Y_2O_3,SiO_2, analogous to gadolinite, is obtained in microscopic, highly bi-refractive, monoclinic crystals, which are attacked by acids. Some measurements of the faces and angles are given. The artificial crystals differ from those of gadolinite in their angle of extinction and in the separation of the optical axes, the differences being doubtless due to the presence in gadolinite of oxides other than yttria.

In the preparation of yttrium silicates by Lechartier's method, a mixture of cubes and elongated prisms is obtained, and if this is fused with sodium carbonate and the product treated with acids, the prisms, which consist of a double silicate of calcium and yttrium, are decomposed and dissolved, whilst the cubic crystals which consist of yttrium oxide remain unaltered.

When amorphous yttrium oxide is heated to redness with calcium chloride for about two hours, and the product is extracted with warm water, highly refractive crystals of yttrium oxide are obtained, and these are attacked with difficulty by acids or by fused sodium

carbonate. The crystals are trapezohedrons resembling those of analcime, but they have no action on polarised light.

Yttrium oxide heated to redness on a gas-carbon support in a current of chlorine and carbonic oxide yields yttrium chloride in confused lamellæ, very soluble in water, with formation of the very hygroscopic hydrate $Y_2Cl_6 + 12H_2O$, described by Cleve. In the preparation of the chloride, care must be taken that the current of gas is not too rapid, otherwise a considerable quantity of the salt is volatilised. If the tube is heated to 1100°, the chloride melts, and distinct crystals of considerable size can be observed floating on the surface of the liquid. The crystals are not affected by benzene. They are transparent, colourless, highly bi-refractive, rectangular lamellæ with transversal cleavage. The bisectrix is negative, and the separation of the optical axes is about 50°.

Yttrium bromide is obtained in a similar manner. It closely resembles the chloride in appearance, is very deliquescent, and with water forms the hydrate $Y_2Br_6 + 18H_2O$, described by Cleve.

If dry hydrogen sulphide is passed over a mixture of anhydrous yttrium chloride with excess of sodium chloride, heated at 1000° in a boat of gas-carbon, a greenish, crystalline product is obtained, and when this is treated with water, transparent, greenish lamellæ of the compound, Na_2S,Y_2S_3, are left undissolved. The crystals do not alter at the ordinary temperature, but burn if heated to redness. They are not affected by warm water, but are dissolved even by dilute acetic acid. The form of the crystals is a hexagonal prism flattened in a direction perpendicular with the principal axis, with three directions of cleavage parallel with the sides of the basal hexagon, but the symmetry of the crystals is not perfect, owing to the presence of impurities. C. H. B.

Production of Metallic Chlorides from Oxides. By A. Faure (*Compt. rend.*, **107**, 339—340).—The oxide, such as bauxite, is heated directly by means of the flame from a regenerative furnace, and when the temperature is sufficiently high, the flame is cut off, and a mixture of hydrogen chloride and naphthalene vapour is passed into the heated material. These gases are completely utilised in the conversion of the oxide into chloride, and have little or no injurious action on the bricks of the furnace.

The mixture of hydrogen chloride and naphthalene alone is not decomposed even at a bright white heat, or at any rate no substance is formed which condenses at temperatures above 100°.

C. H. B.

Vapour-density of Gallium Chloride. By C. Friedel and J. M. Crafts (*Compt. rend.*, **107**, 306—309).—Carefully sublimed gallium chloride was volatilised in an atmosphere of nitrogen. Direct experiment proved that no chlorine was liberated. The following results were obtained:—

Temperature	237°	307°	357·15°	377·6°
Vapour-density ..	11·73	10·61	9·08	7·82

The values previously obtained by L. de Boisbaudran were—

Temperature	247°	273°	357°	440°
Vapour-density ..	13·4	11·9	10·0	7·8

It follows that up to 273° gallium chloride has the formula Ga_2Cl_6, but beyond this temperature the molecule dissociates into $2GaCl_3$. (Compare Nilson and Petterssen, Trans., 1888, 824.) In the group containing aluminium, gallium, indium, the tendency of the chloride to dissociate into simpler molecules at higher temperatures increases as the atomic weight increases. The gallium chloride employed by the authors was not absolutely dry, and hence the values obtained are somewhat too low.

Experiments by the diffusion method prove that when aluminium chloride is strongly heated in an atmosphere of nitrogen, no chlorine is liberated, and hence the decrease in vapour-density must be due to the dissociation of Al_2Cl_6 into $2AlCl_3$. (Compare this vol., p. 1040.)

C. H. B.

Vapour-density of Chlorine and Ferric Chloride. By C. Friedel and J. M. Crafts (*Compt. rend.*, **107**, 301—306).—The authors have previously shown (this vol., p. 1040) that the vapour-density of aluminium chloride is constant between 288°, and 400° and corresponds with the formula Al_2Cl_6. Deville and Troost's experiments with ferric chloride gave results corresponding with the formula Fe_2Cl_6, but the later determinations of V. Meyer and Grünewald have shown that between 440° and 1300° the vapour-density is always lower than that corresponding with Fe_2Cl_6, and agrees more nearly with the formula $FeCl_3$. There is always, however, a want of agreement between the observed and calculated values, because at temperatures above 518° the ferric chloride decomposes into ferrous chloride and chlorine. The authors point out that Meyer's results do not agree at all well with the formula $FeCl_3$, and consider that above 750° the ferric chloride dissociates into Fe_2Cl_4 and Cl_2, whilst the further reduction of density observed above 1052° is due to the partial dissociation of Fe_2Cl_4 into $2FeCl_2$. The boiling point of ferrous chloride and its vapour-density are, however, not yet known.

The modification of Dumas' apparatus employed by the authors makes it easy to ascertain whether dissociation has taken place and any crystals have been deposited. They find that, contrary to the statement of Meyer and Grünewald, ferric chloride in an atmosphere of nitrogen is dissociated into chlorine and ferrous chloride, the latter being deposited in almost colourless crystals which do not recombine with the liberated chlorine on cooling, nor after remaining in contact with it at the ordinary temperature for several days. The ferrous chloride is not volatile at 440°, and hence the volume of gas in the apparatus is not altered in consequence of the dissociation.

In order to prevent the dissociation, the apparatus was filled with chlorine, and hence it became necessary to determine the relative density of this gas at the temperatures employed. The chlorine was made in some experiments from manganese peroxide and hydrochloric acid, in others from potassium dichromate and hydrochloric

acid, and was dried by means of sulphuric acid. The following results were obtained:—

Temperature	19·7°	21·6°	23·0°	356·9°	440·0°
Sp. gr.....	2·479	2·458	2·475	2·451	2·448

Between 21° and 357° the coefficient of expansion of chlorine is to that of air as 1·009 : 1·000.

The experiments with ferric chloride were made in the same way as those with aluminium chloride. Relatively low temperatures were employed, and the quantity of the salt used was such that chlorine was always present in considerable excess. The ferric chloride volatilised completely and not a trace of ferrous chloride was formed. Ferric chloride boils at 280—285°, and melts under pressure at 301°. The following results were obtained:—

Temperature ..	321·6°	325·2°	356·9°	357·0°	442.2°	442·2°
Vapour-density.	11·41	12·47	12·04	11·85	11·66	11·30

Between 321° and 442° the vapour-density is practically constant, and corresponds with the formula Fe_2Cl_6. C. H. B.

Action of Potassium Nitrite on Ferric Chloride. By L. Pesci (*Chem. Centr.*, 1888, 1023—1024, from *Ann. Chim. Farm.* [4], **7**, 305—307).—Ferric chloride and potassium nitrite react together with evolution of nitric oxide, whilst a brownish-red precipitate forms. In a flask, from which the air had been displaced by means of carbonic anhydride, a solution of potassic nitrite was introduced and then one of ferric chloride. After the evolution of nitric oxide ceased, the colourless liquid was separated from the brown precipitate, and the excess of nitrous acid destroyed by carbamide and acetic acid, and finally concentrated sulphuric acid was added, when the presence of nitric acid was ascertained. The reaction may probably be represented by the following equation:—

$$Fe_2Cl_6 + 6KNO_2 + 2H_2O = Fe_2O_2(HO)_2 + 6KCl + 2HNO_3 + 4NO.$$

The brown precipitate of the reaction, probably metaferric hydrate, is best separated by dialysis. It is soluble in water, and behaves entirely differently from the ferric salts. With ferrocyanide, a brown coloration is first formed, which eventually develops into a precipitate. Ferricyanide and thiocyanate give no precipitate. Tannin gradually produces a brown precipitate. A trace of a salt of alkali or sulphuric acid coagulates the oxide immediately. A very small quantity of nitric acid does not precipitate the solution, but the concentrated acid precipitates ferric oxide, which, after some time, gradually dissolves. Carbonic anhydride does not precipitate the oxide. By boiling the solution of this hydrate, a reddish-brown precipitate forms, which, however, redissolves on cooling. Phenolphthaleïn and rosolic acid show an acid reaction in the solution. The solution is very stable.

J. W. L.

Crystallised Hydrated Potassium Ferrite prepared in the Dry Way. By G. Rousseau and J. Bernheim (*Compt. rend.*, **107**,

240—243).—When potassium ferrate is thrown into fused potassium chloride or hydroxide, it decomposes rapidly into potassium ferrite and oxygen. The alkalinity of the flux has no influence on the result as in the case of barium ferrate. If potassium hydroxide is used, the ferrite is not changed by continued heating to bright redness, but if the flux is a mixture of the hydroxide and chloride, the ferrite is gradually converted into reddish, transparent crystals similar to those of sodium ferrite.

When potassium ferrite, obtained by Mitscherlich's method of heating potassium ferric oxalate, is heated to redness with potassium chloride or a mixture of the chloride and hydroxide, it yields red-brown crystals of the same composition as those just described. 5 grams of potassium ferrite fused with 30 grams of potassium chloride and the same quantity of the hydroxide, gave crystals of the composition Fe_2O_3, 86·52; H_2O, 11·34; K_2O, 2·91. When the heating was continued until the greater part of the flux was volatilised, the product had the composition Fe_2O_3, 90·16; H_2O, 5·76; K_2O, 3·73, and hence it is evident that the hydrate first formed was afterwards decomposed.

Better results were obtained by using the semi-crystallised ferrite prepared according to Salm-Horstmar's method of fusing ferric hydroxide with 4 parts of potassium carbonate. This product was mixed with twice its weight of potassium chloride, and carefully heated until most of the latter had volatilised. In this way, comparatively large, transparent, red-brown crystals were obtained. Three different specimens gave the following results:—

Fe_2O_3	85·46	86·34	84·67
H_2O	10·82	11·11	10·94
K_2O	3·57	3·01	3·95

This compound is soluble in mineral acids, but if calcined it acquires an alkaline reaction, and then dissolves slowly even in concentrated boiling hydrochloric acid. It loses 5·08 per cent. of water at 150—160°, 3·45 per cent. at about 300°, and the remainder at a red heat. These facts point to the existence of three distinct hydrates.

If crystallised ferrous sulphate is heated to redness with an equal weight of potassium chloride for half an hour, one of these hydrates is obtained in brilliant lamellæ of the composition Fe_2O_3, 84·33; H_2O, 11·45; K_2O, 3·66. If heating is continued for an hour, the potassium chloride is completely volatilised, and the crystals obtained have the composition Fe_2O_3, 97·20; H_2O, 1·47; K_2O, 1·08. Further heating would doubtless result in the formation of hematite.

It is noteworthy that the water and potassium oxide volatilise simultaneously, a fact which affords further proof that in these compounds they have a similar function. C. H. B.

Chinese Treatment of Cobalt Ores. By T. I. Bowler (*Chem. News*, **58**, 100).—The ore (a sample examined contained per cent., 35 of Fe, 13·1 of Mn, 3·5 of Ni, 2·4 of Co, all as oxides, and 46 per cent. of gangue) is washed, powdered while wet, and allowed to settle in water; the lower layers are then dried, mixed with borax,

and fused up with glass. After 36 or 40 hours, the glass is light blue in colour with a tint of amethyst due to manganese, but colours due to iron or nickel are scarcely apparent: the bottom portion of the melt is worthless. For painting on porcelain, the Chinese frit this same ore with felspar, white kaolin clay, and much borax. D. A. L.

Dissociation of Fused Metallic Sulphides. By T. S. Hunt (*Chem. News*, **58**, 87).—Referring to Moore's note on the separation of a brilliant, ductile, metallic alloy from nickel regulus (Abstr., 1887, 1081), the author points out that he has described the separation of metallic iron from a copper-iron regulus. The fused mass consisted of oxysulphides, and on cooling decomposed into iron, magnetite and sulphides. The separation of copper from rich copper regulus is another instance of this decomposition of sulphides which are only stable at high temperatures. A crystalline subsulphide, Fe_4Ni_2S, is found in cavities in the nickel matte smelting hearth in Pennsylvania. D. A. L.

Zinc Titanates. By L. Lévy (*Compt. rend.*, **107**, 421—423).—A *bibasic titanate*, $TiO_2,2ZnO$, is obtained by heating to bright redness in a brasqued crucible a mixture of 2 parts of titanic oxide, 15 parts of zinc sulphate, and 15 parts of potassium sulphate. It forms black, crystalline grains; sp. gr. at 20° = 4·16. It is decomposed by chlorine but not by hydrogen, and is dissolved by hot and dilute or cold and concentrated acids and alkalis, and also by a fused mixture of potassium nitrate and sodium carbonate. It can also be obtained by heating the sesquititanate with excess of zinc sulphate or choride.

A *sesquititanate*, $2TiO_2,3ZnO$, is obtained by heating to redness a mixture of 1 part of titanic oxide, 5 parts of zinc sulphate, and 1·2 parts of potassium sulphate, and washing the product with dilute sulphuric acid. It forms a yellow, crystalline powder, which is not decomposed by heat. It is decomposed by chlorine or hydrogen, and slowly by warm sulphuric or nitric acid, or a mixture of sulphuric and hydrofluoric acids. It is completely dissolved by cold hydrochloric acid, and is decomposed by fused potash, but not by a mixture of potassium nitrate or sodium carbonate.

The *normal titanate*, TiO_2,ZnO, is obtained in a similar manner from a mixture of titanic oxide, 2 parts; zinc sulphate, 8 parts, and potassium sulphate 3 parts. It is a pale-violet substance with a conchoidal fracture, and not unfrequently forms silky needles; sp. gr. at 20° = 3·17. It is decomposed by chlorine, but not by hydrogen. Boiling acids and concentrated potash attack it with difficulty, but it is slightly attacked by a fused mixture of potassium nitrate and sodium carbonate, and is decomposed by fused potash.

An *acid titanate*, $5TiO_2,4ZnO$, is formed at a cherry-red heat from a mixture of titanic oxide, 3 parts; zinc sulphate, 12 parts, and potassium sulphate, 1 part; the product being washed with dilute nitric acid. It forms small, pale-brown, crystalline leaflets, sp. gr. at 19° = 3·68. It is insoluble in all ordinary solvents, and is decomposed by chlorine, but not by hydrogen. It is not attacked by concentrated acids in the

cold, but is dissolved by all except hydrochloric acid when heated, and is also dissolved by fused potassium hydrogen sulphate.

The nature of the product is determined by the temperature and by the composition of the mixture. At a dull red heat, the sesquititanate is always formed, but at higher temperatures one of the other compounds is formed according to the composition of the mixture, the proportion of potassium sulphate being of great importance. It is probable that a potassium titanate is first formed, but is subsequently decomposed by the zinc salt. In fact, the zinc compounds can be obtained by the direct action of zinc sulphate on potassium titanate. C. H. B.

Fluorine-derivatives of Pertitanic Acid. By A. Piccini (*Gazzetta*, **17**, 479—486).—In a communication to the *Accademia dei Lincei* (Sept., 1885), the author described a barium fluoroxypertitanate, (TiO_2F_2,BaF_2), prepared by adding barium nitrate to a solution of the octahedral ammonium fluoroxypertitanate, when it comes down as a flocculent precipitate. Sometimes also, under conditions which are not accurately known, this was accompanied by a crystalline barium compound, probably $2TiO_2F_2$,$3BaF_2$, corresponding with the octahedral ammonium salt.

When the amorphous barium salt is gently heated, it loses its yellow colour and becomes white, but at a higher temperature it parts with its fluorine, and is converted into a white mass of the composition BaO,TiO_2; the total loss being somewhat more than 20 per cent. of the original weight.

In order to ascertain what was the nature of the reaction which takes place in the first stage, some of the barium fluoroxypertitanate was heated in a vacuum at 150—160°, the gas which was given off collected, and the residue weighed. The gas evolved was pure oxygen, and the residue had the composition $TiOF_4Ba$, the decomposition taking place according to the equation $2(TiO_2F_2,BaF_2) = 2TiOF_4Ba + O_2$. As the residue is easily soluble in dilute acids, it cannot be a mixture containing titanic acid, and is therefore probably a new barium salt of the formula $TiOF_2,BaF_2$.

Fluoroxypertitanic acid, $TiO_2F_2,2HF$, may be obtained in solution by adding barium fluoroxypertitanate, in slight excess, to well-cooled dilute sulphuric acid (4—5 per cent.) contained in a platinum dish, filtering (using a silver funnel) from the barium sulphate and excess of fluoroxypertitanate, and cautiously precipitating the traces of barium in solution with dilute sulphuric acid. If an alkaline carbonate is added to the liquid, it effervesces and deposits yellow peroxide of titanium, but on cautiously neutralising with potassium hydroxide, a crystalline precipitate of potassium fluoroxypertitanate, $TiO_2F_2,2KF$, is obtained; the ammonium salt could not be obtained definitely crystallised. On saturating the acid with ammonia and adding ammonium fluoride, the octahedral ammonium fluoroxypertitanate, $TiO_2F_2,3NH_4F$, is thrown down in the crystalline state. That the solution really contained an acid of the formula given above was confirmed by determining the relation between the titanium and fluorine.

The author has been unable to obtain any zirconium compounds corresponding with those of titanium just described. C. E. G.

Amorphous Antimony. By F. Hérard (*Compt. rend.*, **107**, 420).—When antimony is heated to dull redness in a current of nitrogen, greyish vapours are evolved, which condense on the cool parts of the tube in agglomerations of small spheres similar in appearance to Bettendorff's amorphous arsenic. The sublimate contains 98·7 per cent. of antimony, and is similar to the amorphous antimony obtained by Gore by the electrolysis of the haloïd salts. It melts at 614°; sp. gr. at 0° = 6·22. Ordinary crystallised antimony melts at 440°; sp. gr. = 6·73. The fact that the amorphous variety is not produced in a current of hydrogen seems to indicate that a nitride is first formed, but decomposes in the cooler parts of the tube. C. H. B.

Bismuth and Lithium in Iron and Slags. By H. N. Warren (*Chem. News*, **58**, 27).—Hydrogen sulphide produced a decided black precipitate of bismuth sulphide in hydrochloric acid solutions containing 5 grams of some Cleveland district ordinary blast slags, in six samples out of nine. Estimated in the usual way, the amount was from 0·1 to 0·5 per cent. of bismuth oxide in the different samples; the corresponding samples of iron contained 0·01 to 0·04 per cent. of metallic bismuth. The slags were also found to contain from 0·05 to 0·01 per cent. of lithium estimated as carbonate, which was also detected spectroscopically in the residue obtained from four samples of cast iron out of six, after removal of the iron and other substances. D. A. L.

Halogen Compounds of Gold. By G. Krüss and F. W. Schmidt (*J. pr. Chem.* [2], **38**, 77—84; compare this vol., p. 28).—The authors have again failed to obtain a chloride of gold having the constant composition Au_2Cl_4, notwithstanding that they have exactly followed Thomsen's latest directions (this vol., p. 559). The product of the action of chlorine on gold is a mixture of auric chloride and unaltered gold, for the former may be dissolved away from the latter by water and by ether. Nor is the compound Au_2Br_4 obtained by the action of bromine on gold: when the absorption of bromine ceases, the product is auric bromide mixed with a trace of unaltered gold. Only a small quantity of the gold can be converted into auric bromide by bromine vapour. A. G. B.

Rhodium Salts. By E. Leidié (*Compt. rend.*, **107**, 234—237).—If a saturated solution of ammonium chloride is added to a concentrated solution of ammonium rhodium chloride containing a considerable proportion of free nitric acid, crystalline plates resembling chromic chloride are deposited. When these are dried in a vacuum, they have the composition $Rh_2Cl_6,6NH_4Cl,2NH_4NO_3$. If treated with water, they decompose into the double chloride $Rh_2Cl_6,6NH_4Cl + 3H_2O$, rhodium chloride, and gaseous products similar to those produced by the action of aqua regia on ammonium chloride. The feeble solubility of this compound must be borne in mind when ammonium chloride is employed to separate rhodium from platinum.

Rhodium sulphate, $Rh_2(SO_4)_3$, is obtained by dissolving rhodium sesquioxide in excess of sulphuric acid, evaporating to a syrup, and finally heating at 440° until the weight is constant. At this temperature, the whole of the free acid is expelled, but the sulphate is not decomposed. It forms a brick-red, non-hygroscopic powder, stable in presence of water if the latter does not exceed 16 equivalents per equivalent of salt, but decomposed by a larger quantity with formation of a basic sulphate and free sulphuric acid. When boiled with large quantities of water until the washings are no longer acid, it yields a subsulphate, $Rh_2(SO_4)_3,Rh_2O_3$. Rhodium sulphate forms no alums and no crystallisable double sulphates.

The *double oxalates* have the general formula $Rh_2(C_2O_4)_3,3M_2C_2O_4 + nH_2O$, crystallise readily, and are analogous to but not isomorphous with the double ferric and chromic oxalates. *Rhodium potassium oxalate* is obtained in garnet-red, triclinic prisms, very soluble in water, by dissolving freshly precipitated rhodium sesquioxide in a solution of potassium hydrogen oxalate and crystallising in a vacuum. In a concentrated solution, the ordinary reactions of rhodium and oxalic acid are partially masked; in dilute solutions, the salt is decomposed with precipitation of rhodium sesquioxide. The sodium and ammonium salts have similar properties, and are obtained in a similar manner. The former crystallises with 12 mols. and the latter with 9 mols. H_2O. *Rhodium barium oxalate*, obtained by the action of barium chloride on the potassium salt, forms orange-yellow crystals, which contain 6 mols. H_2O and are only slightly soluble in boiling water. C. H. B.

Note by Abstractor.—The fact that in solutions of these so-called double oxalates the reactions of rhodium and oxalic acid are partially masked seems to indicate that these compounds are really rhodio-oxalates, analogous in constitution to the chromoxalates.

C. H. B.

Mineralogical Chemistry.

Cobalt Ores. By G. Vollhardt (*Zeit. Kryst. Min.*, **14**, 407—408).—Crystals of smaltine and cloanthite when cut and treated with acids exhibit a series of zones of varying solubility. In order to determine whether this is due to varying composition, specimens of ore were powdered and treated with hydrochloric acid and potassium chlorate. A portion was thus dissolved, and of the residue a portion was used for analysis and a portion treated in the same way, the final residue being analysed. The results were as follows:—

	Ia.	Ib.	Ic.	Id.	IIa.	IIb.	IIIa.	IIIb.
SiO_2.....	0·14	0·12	0·13	0·16	—	—	—	—
S	0·61	0·61	(0·61)	(0·61)	0·30	(0·30)	—	—
Bi.....	0·31	0·41	0·16	0·18	4·58	0·37	—	—
Pb	0·37	0·36	0·01	0·12	—	—	—	—
As	75·53	73·46	75·78	76·19	71·19	75·43	77·94	78·26
Fe	0·37	0·38	0·47	0·30	1·35	1·22	5·07	6·69
Ni ..... }	21·94	{ 19·88	19·89 }	21·71	21·84	22·24	12·01 }	15·05
Co ..... }		{ 2·03	2·30 }				3·69 }	
Totals	99·27	97·25	99·35	99·27	—	—	—	—

I. Cloanthite from Schneeberg in Saxony: *a* and *b*, original ore; *c*, first residue; *d*, second residue. II. Cloanthite crystals, cubes with pentagonal dodecahedra, from Wolkenstein in Saxony: *a*, original ore; *b*, residue. III. Cloanthite from Markirch in Alsace, sp. gr. 6·32.

The results obtained with the Schneeberg cloanthite (I), showing that for the most part a compound poor in arsenic has been dissolved, are confirmed by the fact that the compound $CoAs_3$ (skutterudite) is soluble with considerably greater difficulty than smaltine and cloanthite. Whether an admixture of RAs_3 or of free arsenic is present in the latter minerals cannot be determined. In the cloanthite from Wolkenstein (II) the ratio of R to As has increased from 1 : 2·4, in the residue to 1 : 2·62. The bismuth appears not to be in combination with the metals. The Alsatian cloanthite (III) when treated with acid was found to be homogeneous. It has the formula RAs_3, whilst other crystals from the same locality previously analysed gave the formula RAs_2. The author concludes that further investigations are necessary to determine the chemical composition of smaltine and cloanthite. B. H. B.

Calciostrontianite (Emmonite) from Brixlegg. By A. Cathrein (*Zeit. Kryst. Min.*, **14**, 366—374).—A mineral occurring in small, well-developed, rhombic crystals on barytes in the Grosskogel grey copper mine at Brixlegg in the Tyrol, was first thought to be smithsonite, and subsequently strontiocalcite. It gave on analysis the following results:—

$SrCO_3$.	$CaCO_3$.	Total.
86·89	13·14	100·03

The mineral is therefore a calciostrontianite having the formula $9SrCO_3,2CaCO_3$. It is identical with the American mineral named emmonite by T. Thomson in 1836. As the barytes matrix contains 0·71 per cent. of strontium sulphate and 0·1 per cent. of calcium sulphate, the calciostrontianite may have been formed from this by the action of water containing carbonic acid. B. H. B.

Phosphorite of Capo di Leuca. By I. Giglioli (*Gazzetta*, **18**, 54—56).—This phosphorite occurs abundantly in the pliocene limestone of Capo di Santa Maria di Leuca, at Otranto. It is found in nodules and fragments, which are very hard, and can readily be separated from the calcareous gangue by calcining the lumps in a furnace and then treating them with cold water. As yet no technical use has been made of this bed of phosphorite.

The results of the analysis are as follows:—

$Ca_3(PO_4)_2$.	$CaCO_3$.	$CaSO_4$.	Fe_2O_3.	Silica.	Al_2O_3, &c., by difference.	H_2O.
39·22	39·84	1·81	4·02	3·88	12·98	1·25

C. E. G.

Arnimite. By A. Weisbach (*Zeit. Kryst. Min.*, **14**, 399—400, from *Jahrb. Berg. Hüttenw. K. Sachsen*, 1886, 86).—The name of arnimite is applied by the author to a mineral occurring in the so-called porcelain-jasper of Planitz near Zwickau. Under the microscope, this mineral is seen to be an aggregate of short, acicular crystals. A specimen, as free as possible from the accompanying anhydride and gypsum, gave on analysis the following results (I):—

	CuO.	SO_3.	Fe_2O_3, Al_2O_3.	CaO.	H_2O.	Total.
I.	56·81	24·43	0·35	0·56	(17·85)	100·00
II.	49·96	24·59	—	8·17	17·76	100·48

These results are in fair accord with the formula $Cu_5S_2O_{11} + 6H_2O$. As this composition approaches most nearly to that of herrengrundite, a new analysis was made of that Hungarian mineral, the results of which (II) lead to the formula $Cu_4CaS_2O_{11} + 6H_2O$, showing that herrengrundite is a lime-bearing arnimite. B. H. B.

Minerals of the Tetragonal System present in Crystallised Slags. By J. H. L. Vogt (*Chem. Centr.*, 1888, 985, from *Berg. Hütt. Zeit.*, **47**, 187—189).—The author contends that his former assertion concerning the identity of certain crystalline minerals found in slags with the natural melilite and gehlenite is supported by the optical properties and structure of the minerals. He has also noticed in the blast furnace slags another mineral in which lime is the principal base, apparently closely related to melilite. J. W. L.

Alteration of Quartz into Talc. By E. Weinschenk (*Zeit. Kryst. Min.*, **14**, 305—323).—The extensive talc deposit of Göpfersgrün in the Fichtelgebirge occurs at the contact of granite and crystalline schists, consisting of mica-schist, phyllite, and, more rarely, serpentine, variolite, and chlorite-schist. It forms part of a limestone bed in which occur quartz in crystals and grains, fluorspar, chondrodite, magnetic pyrites, iron pyrites, garnet, phlogopite, grammatite, graphite, and many other minerals. The talc deposited on the limestone contains pseudomorphs of talc after brown spar and quartz, and more rarely of talc after calcite, and of chalcedony after dolomite. The following analyses indicate the purity of the talc:—

	SiO_2.	MgO.	Fe_2O_3.	H_2O.	Total.
I.	63·52	31·72	—	4·76	100·00
II.	62·87	31·62	1·31	3·93	99·73
III.	63·32	31·49	0·57	4·38	99·76
IV.	62·98	31·36	1·85	4·32	100·51

I is the composition of pure talc calculated from the formula $Mg_3(SiO_3)_4H_2$; II is the composition of a yellowish-white pseudomorph after quartz; III of a pure white dolomite pseudomorph, and IV of ordinary greenish talc.

The process of the alteration of the quartz into talc was elucidated by a microscopical examination of the quartz occurring in the rock immediately above the talc bed, where every stage of the transition can be distinctly seen. B. H. B.

Artificial Magnesia Mica. By J. H. L. Vogt (*Chem. Centr.*, 1888, 985, from *Berg. Hütt. Zeit.*, **47**, 197—199).—A magnesia mica nearly always crystallises out of the copper slag of the Kafveltorp copper works in Sweden, which is but slightly different from the natural mineral, and has a composition agreeing with the proportions Si : Al : Mg(+Fe) : K(+Na) = 1·017 : 0·336 : 1·00 : 0·384, whereas these are 1 : 0·4 : 1 : 0·4 in the natural mineral. It would appear that the presence of fluoride in the slag is a necessary condition to the formation of the mica. J. W. L.

Biotite from Christiania. By P. Jannasch (*Zeit. Kryst. Min.*, **14**, 400).—In a Norwegian journal, the author gives the following analysis of the brown biotite from the micaceous rock that forms a portion of the contact zone in the Silurian basin of Christiania :—

SiO_2.	TiO_2.	Nb_2O_5.	Al_2O_3.	FeO.	MgO.	CaO.	Na_2O.	K_2O.	H_2O.	Total.
33·95	3·40	0·98	17·69	21·94	7·98	1·10	1·00	8·39	3·46	99·89

Traces of manganese, strontium, and lithium were also observed. The analytical results correspond with the empirical formula $R'_{18}R''_{15}R'''_{10}Si_{18}O_{75}$. B. H. B.

Cronstedtite from Kuttenberg in Bohemia. By K. Vrba (*Zeit. Kryst. Min.*, **14**, 410, from *Sitzber. Böhm. Ges. Wiss.*, 1886).—At Kuttenberg, cronstedtite has been found in black, fibrous masses on cryptocrystalline, opaque quartz, or on a granular mixture of quartz, pyrites, and blende. The mineral has a sp. gr. of 3·445, and on analysis gave the following results :—

SiO_2.	Fe_2O_3.	FeO.	MnO.	H_2O.	Total.
17·34	43·05	30·27	0·16	(9·18)	100·00

Formula, $H_8Fe''_3Fe'''_4Si_2O_{17}$. B. H. B.

New Manganese Ore from Dillenburg. By Schneider (*Chem. Centr.*, 1888, 1016, from *Zeit. deut. geol. Ges.*, **39**, 829—834).—This ore, which was struck in 1885, is a lode of 0·60—1·0 m. thick.

Its colour varies from light brown to red and dark brown. The crystalline form could not be determined. Hardness 6—7; sp. gr. 3·1026. Analysis gave—

SiO_2.	MnO.	CaO.	H_2O.	Al_2O_3.	FeO.	MgO.	Total.
43·92	38·23	8·00	8·49	0·29	0·69	0·28	99·90

It has been named "Inesite." J. W. L.

Analysis of the Chalybeate Water of Raffanelo in the Commune of Canale Monterano. By F. Mangini (*Gazzetta*, **17**, 517—519).—The spring rises from compact, calcareous rocks interspersed with crystals of calcite, the yield being about 3000 litres per diem. The density at 15° is 0·9745. The water contains 436 c.c. of carbonic anhydride, and the iron, present as ferrous carbonate, is equal to 0·0220 gram per litre; it contains neither nitrates nor nitrites. The solid residue per litre dried at 110° = 1·0915 gram.

The composition of this residue is as follows:—

Carbonic anhydride, CO_2	0·4240
Silica, SiO_2	0·0279
Sulphuric acid, H_2SO_4	0·0820
Phosphoric acid, H_3PO_4	0·0014
Chlorine, Cl	0·0053
Iodine	traces
Ferrous oxide, FeO	0·0089
Calcium oxide, CaO	0·3343
Magnesium oxide, MgO	0·0148
Aluminium oxide, Al_2O_3	0·1350
Potassium oxide, K_2O	0·0369
Sodium oxide, Na_2O	0·0100
Loss	0·0110

C. E. G.

Water of the Nile. By A. Muntz (*Compt. rend.*, **107**, 231—234). Estimations of the quantity of nitrates in the water of the Nile gave the following results, which are expressed in milligrams per litre:—

	July, 1887.	August, 1887.	September, 1887.
Nitric acid	4·09	4·02	1·83
Equivalent to—			
Potassium nitrate	7·62	7·51	3·71
Or nitrogen	1·067	1·052	0·519

It is evident that the quantity of nitrates is very variable and does not exceed that found in many ordinary rivers. The nitrates are derived partly from the atmosphere and partly from the soil. In tropical districts the proportion of nitric acid in the rain-water is much more considerable than in temperate climates. The fertility of the district watered by the Nile cannot be attributed to the nitrates contained in the water, but in all probability is due to the constituents of the fine mud which the water deposits. C. H. B.

Organic Chemistry.

Ethyl Fluoride. By H. Moissan (*Compt. rend.*, **107**, 260—263).—Ethyl iodide is allowed to drop gradually on to silver fluoride in a brass vessel, care being taken that the temperature does not rise too high. Any volatilised ethyl iodide is condensed by means of a serpentine lead tube cooled to −20°, and is allowed to fall back upon the silver salt. The last traces of the iodide are removed from the gas by passing it through two U-tubes containing silver fluoride. It is then collected in carefully dried glass vessels over mercury. Ethyl iodide in contact with excess of silver fluoride develops heat, and a gas is evolved, whilst at the same time the yellow colour of the fluoride changes to maroon. Under these conditions, a silver fluoriodide is formed, and this in contact with a fresh quantity of ethyl iodide at 100° yields silver iodide and ethyl fluoride.

Ethyl fluoride is a colourless gas with an agreeable ethereal odour; sp. gr. 1·70. It liquefies at −48° under normal pressure or under 8 atmos. at 19°, and can be solidified by suddenly diminishing the pressure. It dissolves readily in several liquids. Water dissolves 1·98 vols. at 14°; ethyl iodide dissolves 14·8 vols.; ethyl bromide, ether, and absolute alcohol especially, also dissolve large quantities. When the liquids are heated, the gas is completely expelled unaltered. Concentrated sulphuric acid also dissolves the gas.

Ethyl fluoride burns with a blue flame which in presence of small quantities of methyl or ethyl chlorides becomes green. Admixture with a small quantity of oxygen produces a bright flame, and with an excess of oxygen the gas explodes violently when ignited. When heated in sealed tubes at 100° with very dilute potash, ethyl fluoride is completely decomposed, and yields potassium fluoride, alcohol, and a large proportion of ether.

Chlorine has no action on ethyl fluoride in the dark after several hours, but gaseous fluorine completely and rapidly decomposes ethyl chloride. It follows that fluorine displaces chlorine from organic as well as inorganic compounds. C. H. B.

Propylene Iodide from Allyl Iodide and Hydrogen Iodide. By H. Malbot (*Compt. rend.*, **107**, 113—115).—If allyl iodide, cooled at −18°, is treated with a regular current of hydrogen iodide, it yields propylene iodide; but before combination is complete, the latter changes into isopropyl iodide without evolution of propylene. The change takes place slowly, but if the liquid is treated with a further quantity of hydrogen iodide, the change becomes complete.

If the allyl iodide is not cooled, the propylene iodide may decompose very violently: if it is heated, the propylene iodide decomposes as fast as it is formed. When the allyl iodide is mixed with isopropyl iodide, the decomposition of the propylene iodide takes place less rapidly, but still with considerable violence. This constitutes a convenient method for the preparation of propylene. If allyl iodide

is saturaled with hydrogen iodide and heated in sealed tubes at 100°, it is completely converted into isopropyl iodide.

In the preparation of isopropyl iodide by Markownikoff's method, if only 10 per cent. of water is added to the glycerol, a very violent reaction takes place, even with red phosphorus, and propylene iodide is formed in large quantity. The formation of propylene iodide cannot be avoided by using anhydrous glycerol, since some water is formed in the reaction. C. H. B.

Chemistry of Combustion. By C. Lüdeking (*Annalen*, **247**, 122—128).—The author finds that the characteristic red compound of titanium cyanide and nitride, $3Ti_3N_2 + Ti(CN)_2$, is invariably formed when a small quantity of titanic acid and sodium carbonate contained on a loop of a platinum wire is introduced into the flame of a candle or of a spirit-, petroleum-, or paraffin-lamp. The cyanogen formed by burning 50 litres of methane in air free from ammonia can be detected by means of the prussian-blue reaction. Hence it appears that nitrogen unites directly with carbon at the temperature of the flame of an ordinary candle or spirit-lamp. W. C. W.

Influence of Temperature on the Production of Higher Alcohols by Fermentation. By L. Lindet (*Compt. rend.*, **107**, 182—183).—Worts prepared by the action of malt on maize and rye were fermented at different temperatures by equal quantities of the same mixture of high and low yeast, the access of air to the liquids being prevented. The alcohols were separated and analysed, and the following table gives the quantities obtained from 30 litres of wort:—

		Higher alcohols.	
Temperature.	Crude alcohol.	Total.	per cent.
32—35°	675 c.c.	3·9	0·58
25—27	1607 „	9·6	0·59
19—21	1834 „	9·9	0·54
8—10	1877 „	9·7	0·52

It is evident that although the differences are greater than can be fairly attributed to experimental error, the proportion of the higher alcohols is not greatly reduced when fermentation takes place at a low temperature. C. H. B.

Fusel Oil in Beer. By W. M. Hamlet (*Chem. News*, **58**, 81—83, 87—88).—Fusel oil to the extent of from a quarter to one grain per gallon has been found in beer in Sydney, N.S.W., by the following method of analysis. A gallon of beer is shaken by instalments with chloroform, 50 c.c. being used for the first half gallon, another 50 c.c. is added and used with the next pint, finally 50 c.c. more is added, making in all 150 c.c. of chloroform, and the rest of the gallon of beer extracted. The extract is washed with water to remove ethyl alcohol and any valeral from the hops, it is then heated under pressure for six hours at 85°, with 5 grams of potassium dichromate and 2 grams of concentrated sulphuric acid, finally it is distilled; the residue mixed

with water and distillation continued. The distillate has a strong odour of valeric acid; it is boiled with barium carbonate, the chloroform distilled off, and the quantity of barium valerate determined and attributed to fusel oil. If the chloroform extract washed with water does not yield valeric acid, whilst the unwashed chloroform extract does, the author accepts it as an indication that hops have been employed, the valeric acid originating in the latter case from the valeral of the hop oil. D. A. L.

Oxidation of Glycerol. By E. Fischer and J. Tafel (*Ber.*, **21**, 2634—2637. Compare this vol., p. 358).—Almost pure glycerose can be prepared by treating lead glyceroxide with dry bromine, but the success of the experiment depends greatly on the state of aggregation of the glyceroxide. Lead glyceroxide is prepared as follows: dry finely-divided lead hydroxide (500 grams) is added to boiling, aqueous glycerol (1000 grams), containing about 85 per cent. of glycerol, the mixture stirred until it becomes homogeneous, cooled quickly in ice-cold water with constant stirring, and finally 2½ litres of ice-cold alcohol added. The glyceroxide is washed by shaking with alcohol and ether and dried at 100°. The yield is 85 per cent. of the hydroxide employed, and the product contains small quantities of nitric acid. It explodes when heated, and takes fire when placed in contact with chlorine or bromine. (Compare Morawski, Abstr., 1880, 428.)

For the preparation of glycerose, the lead glyceroxide is spread in thin layers and kept under a bell-jar, in proximity to half its weight of bromine contained in a shallow dish, until the bromine is entirely absorbed. The product is extracted with alcohol, the solution quickly concentrated on the water-bath in a partial vacuum, and mixed with barium carbonate to remove free acid. On shaking with ether, the greater part of the glycerose passes into solution, and the remainder can be obtained from the residue by extracting with warm alcohol, precipitating with ether, and repeating the operation. The ether is evaporated from the combined extracts, water added and the solution concentrated on the water-bath in a partial vacuum.

The syrup thus obtained contains traces only of inorganic matter and is free from glycerol and acids. The reducing power of a syrup prepared from 860 grams of the glyceroxide is equal to that of a solution containing 55 grams of grape-sugar, but the reducing power is greatly diminished when the syrup is evaporated to dryness, probably owing to polymerisation. The diluted syrup ferments with yeast, and when warmed with phenylhydrazine, phenylglycerosazone is precipitated. It yields two hydroxy-acids when warmed with concentrated hydrocyanic acid, showing that glycerose is a mixture. These acids, which can be separated by means of their barium salts, are probably erythroglucic acid and *trihydroxyisobutyric* acid. F. S. K.

Polybasic Glyceroxides. By de Forcrand (*Compt. rend.*, **107**, 269—272).—The heat of neutralisation of glycerol by three successive equivalents of sodium hydroxide at 20° develops +0·50, +0·14 and +0·04 Cal. respectively. The corresponding values in the case of potassium hydroxide being +0·56, +0·14, +0·09.

When monopotassium glyceroxide methylate, $C_3H_7O_3K + MeOH$, is mixed with an equivalent quantity of potassium methoxide dissolved in methyl alcohol, the liquid evaporated to dryness, and the residue heated in an atmosphere of hydrogen for several hours at 180°, the product is not dipotassium glyceroxide, but a compound of monopotassium glyceroxide with the methoxide, analogous to the sodium compound previously obtained under similar conditions. It would seem that the combination of an alkaline monoglyceroxide with the corresponding alkyl oxide is a general reaction.

Attempts to prepare trisodium glyceroxide in a similar manner were equally fruitless. C. H. B.

Action of Ammonia on Epichlorhydrin. By A. Fauconnier (*Compt. rend.*, **107**, 115—117).—When a current of ammonia gas is passed through epichlorhydrin at the ordinary temperature there is no appreciable action, but if the two are left in contact for some time, the gas is slowly absorbed and the liquid gradually becomes viscous. Towards the close of the reaction, a considerable quantity of heat is developed, and it is necessary to cool the liquid in order to prevent carbonisation. When elevation of temperature is prevented, the liquid is converted into a white mass of microscopic, truncated octahedra. These are washed with cold alcohol, dissolved in dilute acetic acid, and the liquid neutralised with ammonia, when the crystals separate in their original form. They consist of *tri-chlorhydroxypropylamine*, $N(C_3H_5Cl\cdot OH)_3$, melt at 92—93°, are almost insoluble in cold neutral solvents, and dissolve readily in dilute acids, forming non-crystallisable salts from which the base is precipitated by ammonia. The base alters readily, and if dissolved in hot water or alcohol is converted into a non-crystallisable syrup. The hydrochloride crystallises in needles which melt without alteration at 173°, and are only slightly soluble in alcohol, but somewhat soluble in water. With sodium phosphotungstate, an acetic acid solution of the base yields an amorphous white precipitate; with the phosphomolybdate, an amorphous, lemon-yellow precipitate; with Nessler's reagent, a white amorphous, precipitate; with Bouchardat's reagent, a pale-brown precipitate; but no precipitate with potassium mercuroiodide, potassium cadmioiodide, auric chloride, platinic chloride, mercuric chloride, potassium ferrocyanide, or picric acid. If the alcoholic solution is mixed with silver nitrate, it yields a precipitate of silver chloride in the cold; ammoniacal silver nitrate is slowly reduced in the cold with formation of a mirror.

The action of alkalis on trichlorhydroxypropylamine at the ordinary or a higher temperature converts it into a substance which closely resembles gelatin, and is insoluble in all ordinary reagents and even in boiling concentrated acids. C. H. B.

Mannitol Dibenzoate. By J. Meunier (*Compt. rend.*, **107**, 346—348).—If a solution of mannitol, dehydrated in the manner previously described (this vol., p. 950) and containing a considerable quantity of alcohol not converted into ethyl chloride, is mixed with benzoic chloride, there is considerable development of heat; hydrogen

chloride is evolved and ethyl benzoate is formed. After treatment with alkali to remove zinc, the benzoic chloride is distilled and deposits transparent, monoclinic or triclinic crystals of mannide dibenzoate, $C_{20}H_{22}O_6$, which is very slightly soluble in water or in cold alcohol, but dissolves in boiling alcohol, from which it separates in long prisms melting at 132°. It is soluble in chloroform, glacial acetic acid, sulphuric acid, and benzene. It is not decomposed by dilute or concentrated sulphuric acid, nor by aqueous potash, but is attacked by boiling alcoholic potash, with formation of benzoic acid and some other substance, but no appreciable quantity of mannitol.

In order to avoid the formation of ethyl benzoate in the preparation of this compound, care should be taken that the alcohol is as far as possible converted into ethyl chloride before benzoic chloride is added. After any ethyl benzoate that may have been formed has been expelled by evaporation, the product is washed with cold alcohol to remove a gummy substance, and crystallised from boiling alcohol. C. H. B.

Levulose. By E. Jungfleisch and L. Grimbert (*Compt. rend.*, **107**, 390—393).—Experiments on the inversion of cane-sugar, which will be described in a subsequent paper, confirm the older results, and are at variance with Herzfeld and Winter's statement that saccharose yields 2 mols. of levulose and 1 mol. of glucose.

The levulose employed was purified by crystallisation from alcohol. The rotatory power of a freshly prepared aqueous solution gradually diminishes, but the change is not so strongly marked as in the case of glucose, although, as in that case, it is accelerated by heating. Prolonged heating at temperatures up to 100° causes a reduction in the rotatory power, and concordant results can only be obtained below 40°. The rotatory power of pure levulose varies inversely as the temperature, and increases with the concentration of the solution. The equation $[\alpha]_D = -101{\cdot}38 - 0{\cdot}56t + 0{\cdot}108\ (p - 10)$, where t is the temperature, and p the weight of levulose in 100 c.c., holds good between 0° and 40°, and for all concentrations up to 100°. The rotatory power of pure levulose differs from that calculated from the rotatory power of invert sugar on the assumption that the latter is a mixture of levulose and glucose in equivalent proportions.

C. H. B.

Carbohydrates. By M. Hönig and L. Jesser (*Monatsh.*, **9**, 562—578; compare this vol., p. 246).—The authors have prepared crystallised anhydrous levulose by repeatedly crystallising the pure hydrated substance having the formula $2C_6H_{12}O_6 + H_2O$ from absolute alcohol. At 17·5° it has a sp. gr. = 1·6691. Its specific rotatory power is −113·963 at 20°, and varies both with the concentration of the solution and the temperature. The change in rotatory power owing to variation in the concentration of the solution is represented when $t = 20°$ by the expression—

$$[\alpha]_D^{20} = -113{\cdot}9635 + 0{\cdot}25831q,$$

that due to variation in temperature, by $[\alpha]_D^t = -a + 0{\cdot}67142t$. The reducing power of levulose for all states of concentration less than

1 per cent. is smaller than that of dextrose. The amount of reduced copper, y, obtainable from a particular quantity of the sugar, x, can be calculated by means of the equation—

$$y = -5{\cdot}372 + 1{\cdot}91856x - 0{\cdot}0007605x^2.$$

G. T. M.

Compounds of Phenylhydrazine with Sugars. By E. FISCHER (*Ber.*, **21**, 2631—2634; compare this vol., pp. 39 and 934).—The osazones of all saccharoses dissolve in cold, fuming hydrochloric acid with a dark-red coloration, and the osazone-group, $N_2HPh{\cdot}\dot{C}{\cdot}\dot{C}{\cdot}N_2HPh$, is thereby converted into the group $\cdot CO{\cdot}CO\cdot$.

Oxyglucose, $OH{\cdot}CH_2{\cdot}[CH{\cdot}OH]_3{\cdot}CO{\cdot}COH$, is obtained when phenylglucosazone is mixed with 10 times its weight of ice-cold, fuming hydrochloric acid. The osazone dissolves, and in a short time phenylhydrazine hydrochloride separates; at 5° to 10°, the reaction is at an end in about an hour and a half. The solution is quickly filtered through glass-wool, the residue washed with a small quantity of concentrated hydrochloric acid, the filtrate diluted with ice-cold water, neutralised with lead carbonate, and filtered. The filtrate is warmed with animal charcoal, mixed with excess of baryta-water, and the precipitated lead compound of oxyglucose decomposed with dilute sulphuric acid. After removing the excess of acid, and evaporating in a partial vacuum, the product is obtained in the form of a syrup. The yield is about 30 per cent. of the theoretical quantity. Oxyglucose reduces Fehling's solution on boiling, is precipitated by concentrated baryta-water or a solution of basic lead acetate, and does not ferment with yeast. When an aqueous solution is mixed in the cold with phenylhydrazine acetate, phenylglucosazone separates in a few minutes, but when the mixture is heated at 50—60° the reaction is completed in a very short time, and the osazone obtained is almost pure.

Oxylactose was prepared in like manner from phenyllactosazone, but was not isolated. When the aqueous solution is treated with phenylhydrazine acetate in the cold, phenyllactosazone separates in from 5 to 10 minutes; at 60° to 70°, the formation of the osazone is rapid and complete, and on cooling it crystallises from the solution. Oxyglucose and galactose are formed when the aqueous solution of oxylactose is warmed with hydrochloric acid. From this fact, the author concludes that the constitution of milk-sugar is probably—

$$OH{\cdot}CH_2{\cdot}[CH{\cdot}OH]_4{\cdot}CH\!<\!\begin{matrix}O{\cdot}CH_2\\ \hline O\end{matrix}\!>\!CH{\cdot}[CH{\cdot}OH]_2{\cdot}CH(OH){\cdot}COH,$$

but he does not exclude the alternative possibility, namely, that the two central carbinol-groups (in brackets) take part in the anhydride formation. In accordance with this view, milk-sugar should yield on oxidation an acid, $C_{12}H_{22}O_{13}$, and it was found that when it is treated with bromine in cold aqueous solution a syrupy acid is, in fact, formed. Maltose has probably an analogous constitution. F. S. K.

Vinylamine and Bromethylamine. By S. GABRIEL (*Ber.*, **21**, 2664—2669).—Vinylamine (this vol., p. 668) is best prepared by

treating a cold solution of bromethylamine hydrobromide (1 mol.) with potash (1 mol.), keeping the mixture at 45—48° for 10 minutes in a closed flask, saturating with alkali, and distilling. The last portions of the distillate contain a less volatile base, the *picrate* of which, $C_4H_{12}N_2O,2C_6H_3N_3O_7$, crystallises in yellow needles, melts at 205—206°, and is sparingly soluble in water. For this reason, the distillate should be collected separately as soon as a small portion gives a precipitate with a 1 per cent. solution of picric acid.

This method of preparation depends on the fact that at 45—48° bromethylamine undergoes molecular change into vinylamine hydrobromide.

Hydroxyethylamine nitrate, $OH\cdot CH_2\cdot CH_2\cdot NH_2,HNO_3$, is obtained when bromethylamine nitrate, prepared by mixing equivalent quantities of silver nitrate and bromethylamine hydrobromide in aqueous solution, is boiled with silver nitrate; also, when vinylamine is evaporated with excess of nitric acid. It forms flat, colourless hygroscopic crystals, melts at 52—55°, and explodes on further heating. With sodium picrate, a concentrated aqueous solution gives the salt already described (this vol., p. 440).

When a hot, dilute solution of silver sulphate is mixed with a little less than the molecular proportion of bromethylamine hydrobromide, half the bromine is precipitated as silver bromide, and after boiling for several hours the remainder of the bromine separates as silver bromide when the solution is evaporated. Amidoethylsulphuric acid (*loc. cit.*) is obtained when the solution is freed from silver and concentrated. The constitution of this compound is probably $CH_2<\begin{matrix}CH_2\cdot O\\NH_2\cdot O\end{matrix}>SO_2$.

Vinylamine combines with sulphurous anhydride with formation of taurine.

Freshly prepared vinylamine hydrochloride in doses of 0·03 gram to every kilo. of body-weight proved fatal to guinea-pigs within 10 hours, and doses of 0·015 gram within 24 hours. F. S. K.

Hexamethylenetetramine. By P. Griess and G. Harrow (*Ber.*, **21**, 2737—2739).—*Dinitrosopentamethylenetetramine*, $C_5H_{10}N_4(NO)_2$, is obtained when a solution of sodium nitrite is gradually added to a well-cooled aqueous solution of hexamethylenetetramine containing a slight excess of nitric acid. It crystallises from boiling alcohol, in which it is rather easily soluble, in thick, indented, slightly yellow needles, and from chloroform, in which it is more sparingly soluble, in short, well-defined prisms melting at 207° with decomposition. It has a neutral reaction and a bitter taste, and is almost insoluble in ether and cold alcohol, but dissolves in boiling water with considerable decomposition. In alcoholic alkaline solutions, it is very stable towards reducing agents, but when boiled for a few minutes with very dilute hydrochloric acid it is decomposed, quantitatively and completely, into formaldehyde, ammonia, and nitrogen.

F. S. K.

Ethylenimine. By A. Ladenburg and J. Abel (*Ber.*, **21**, 2706—2707).—The authors have prepared diethylenediamine by treating ethylene chloride with ethylenediamine, and the experiments which

have hitherto been made seem to show that this diamine is not identical with ethylenimine (this vol., p. 441). F. S. K.

Thiazole. By A. HANTZSCH and G. POPP (*Ber.*, **21**, 2582—2583).—*Thiazole*, $N\begin{matrix}CH{:}CH\\CH{\cdot}S\end{matrix}>$, is obtained by boiling the product of the action of nitrous acid on the amidothiazole (formed by the condensation of thiocarbamide and dichlorether) with alcohol. It has an odour similar to that of pyridine, but its salts are distinct from those of that base. N. H. M.

Action of Sodium on Ethyl Chloracetate. By R. FITTIG and A. ERLENBACH (*Ber.*, **21**, 2647—2650).—The compound described as acetoisopropyl alcohol (this vol., p. 1052) is acetol ethyl ether (Henry, *Compt. rend.*, **93**, 421). It yields acetone and ethyl alcohol when treated at 0° with sodium amalgam and dilute hydrochloric acid, and reduces copper acetate in the cold. The *hydrazide*, $C_{11}H_{16}N_2O$, boils at 165°, and when heated at 80—90° with an aqueous alcoholic solution of sodium acetate and sodium chloride, containing a few drops of acetic acid, it yields a colourless, crystalline compound $C_{11}H_{13}NO$, melting at 142·5°, which is probably ethoxymethylindole,

$$C_6H_4\begin{matrix}NH{\cdot}CMe\\C(OEt)\end{matrix}> \text{ or } C_6H_4\begin{matrix}NH\\CH\end{matrix}>C{\cdot}CH_2{\cdot}OEt.$$

F. S. K.

Hydrolysis of Fat. By R. BENEDIKT (*Monatsh.*, **9**, 518—520; compare Abstr., 1884, 946; 1885, 313 and 315; 1887, 620 and 914; and this vol., pp. 578 and 815).—The author finds that the acid obtained by the hydrolysis of fat with sulphuric acid and subsequent distillation with superheated steam, contains neither hydroxystearic nor oleïc acid, but an unsaturated acid, in all probability isoleïc. Commercial stearic acid obtained from fat in the above-mentioned way, is to be regarded as a mixture of stearic, palmitic, and isoleïc acids. G. T. M.

Drying Oils. By A. BAUER and K. HAZURA (*Monatsh.*, **9**, 459—468; compare Abstr., 1887, 359, 798, 913, and this vol., pp. 816, 817).—In this paper, the authors contest many of the views advanced by Mulder on the products of oxidation of drying oils. They find that the rate of oxidation, and consequent hardening, of drying oils, depends on the ratio in which linoleïc and linolenic acids are present; the more linolenic acid there is in the oil, the more rapid is the oxidation, which consists not only in the satisfying of the uncombined valencies with oxygen, but also in the formation of products containing hydroxyl-groups. On oxidation, the salts of the acids behave in a precisely similar manner.

When thin layers of drying acids are left exposed to the air for a considerable period at ordinary temperatures, or for a shorter time at 80°, the product is an anhydride insoluble in ether, but furnishing soluble acids when heated with alkalis. The drying of oils depends on the presence of linoleïc, linolenic, and isolinolenic acids, and in no way on that of oleïc acid, which forms no solid oxidation-products on exposure to air. G. T. M.

Oxidation of Unsaturated Fatty Acids by Permanganate. By K. Hazura (*Monatsh.*, **9**, 469—474).—The author confirms the opinions advanced by Saytzeff (Abstr., 1886, 140) as to the action of permanganate on unsaturated fatty acids. Experiments with the unsaturated acids obtained from poppy-seed oil show that practically no hydroxy-derivatives of fatty acids are formed when permanganate is added to a solution of potassium salts of the acids containing an amount of sulphuric acid just sufficient to neutralise the alkali liberated from the permanganate. On the other hand, when the precaution of adding the sulphuric acid is neglected, the alkali set free on the reduction of the permanganate rapidly brings about the formation of hydroxy-derivatives, a result which is considerably hastened by the addition of free potash. G. T. M.

Acids from Drying Oils. By K. Hazura and A. Grüssner (*Monatsh.*, **9**, 475—484; compare Abstr., 1887, 359, 798, 913, and this vol., pp. 816, 817).—When alkaline solutions of the liquid, fatty acids of castor oil are oxidised with permanganate, *trihydroxystearic acid*, crystallising in microscopic needles melting at 140—142°, and its isomeride, *isotrihydroxystearic acid*, also forming minute needles and melting at 110—111°, are formed, in the ratio of one of the former to two of the latter, together with lower members of the series of fatty acids. Since no dihydroxystearic acid results from the oxidation, no oleïn can be present in castor oil. This is the only observed case in which that constituent is absent in a drying oil. The authors suggest the name *ricinolic acid* for trihydroxystearic acid, and *ricinisolic acid* for its isomeride. G. T. M.

Ricinoleïc Acid. By F. Krafft (*Ber.*, **21**, 2730—2737).—Pure ricinoleïc acid can be prepared as follows:—Castor oil is hydrolysed with concentrated potash, the salt completely decomposed by heating for as short a time as possible with concentrated hydrochloric acid, and the liquid acid, after washing with water, cooled until it solidifies. It is then freed from oily impurities by pressure at a gradually increasing temperature. It is a hard, white, crystalline substance, melts at 16—17°, and solidifies again when cooled considerably below its melting point. The solid acid seems to undergo no change when exposed to the air for several weeks, but in the liquid state it alters slightly. It commences to boil at about 250° (15 mm.), and an oily liquid distils, the temperature rising considerably, but œnanthol and undecylenic acid are not formed. A compound, $C_{18}H_{32}O_2$, boiling at about 230° (15 mm.) is obtained when the distillate is fractionated at 15 mm. pressure. Barium ricinoleate is readily soluble in hot, but only sparingly in cold water. Castor oil which has been kept for a long time in a cool place deposits considerable quantities (3 to 4 per cent.) of a granular, crystalline substance which contains the glycerides of stearic and ricinoleïc acids.

The constitution of ricinoleïc acid is probably

$$CH_3\cdot[CH_2]_5\cdot CH(OH)\cdot CH{:}CH\cdot[CH_2]_8\cdot COOH \text{ or}$$

$CH_3\cdot[CH_2]_5\cdot CO\cdot[CH_2]_{10}\cdot COOH$; in accordance with this view, the

author found that normal heptylic acid is formed, together with azelaïc and oxalic acids, when ricinoleïc acid is oxidised with nitric acid.

Ricinelaïdic acid boils at about 250° (15 mm.) with partial decomposition, and yields normal heptylic acid when treated with nitric acid.

Ricinic acid, $C_{18}H_{34}O_3$, is formed when barium ricinoleate is heated in a partial vacuum; methyl hexyl ketone distils and the acid in the residual salt is precipitated with hydrochloric acid and purified by distillation. It crystallises from alcohol in plates, melts at about 81°, and boils at about 250—252° (15 mm.) with slight decomposition. When treated with nitric acid, it yields principally normal heptylic acid and an acid melting at 100—107°. Towards bromine and potash, it behaves like ricinoleïc and ricinelaïdic acid. F. S. K.

Tetramethylene-derivatives. By M. Freund and E. Gudeman (*Ber.*, **21**, 2692—2699).—*Tetramethylenecarboxylamide*, $C_4H_7{\cdot}CO{\cdot}NH_2$, is obtained by heating ammonium tetramethylenecarboxylate at 230—250° and distilling the product. It crystallises from alcohol in plates, melts at 138°, sublimes unchanged, and is readily soluble in water, ether, chloroform, and benzene. Attempts to convert the amide into the corresponding amine by means of bromine and alkalis were unsuccessful, as were also similar experiments made with the object of preparing carbamide-derivatives. The *nitrile*, $C_4H_7{\cdot}CN$, prepared by heating the amide with phosphoric anhydride and fractionating the product, is a colourless, agreeably smelling liquid boiling at 150°. It is converted into the acid when heated with potash. The ethyl salt, prepared by saturating an alcoholic solution of the acid with hydrogen chloride, boils at 159—162° (compare Perkin, this Journ., 1887, 12). The *chloride*, $C_4H_7{\cdot}COCl$, obtained by boiling the acid with phosphorous chloride, is a fuming liquid boiling at 142—143°. The *anhydride*, $(C_4H_7{\cdot}CO)_2O$, prepared by distilling a mixture of the chloride and the sodium salt, boils at 160°.

Tetramethylenecarbanilide, $C_4H_7{\cdot}CO{\cdot}NHPh$, is formed when the amide is boiled with aniline. It crystallises from alcohol in long, pointed needles, melts at 111° and is sparingly soluble in hot, and insoluble in cold water.

Tetramethylenemethylamine (*tetramethylenylamine*) *hydrochloride*, $C_4H_7{\cdot}CH_2{\cdot}NH_2,HCl$, is prepared by reducing the nitrite with sodium and absolute alcohol, distilling the reaction-product with steam, collecting the distillate in hydrochloric acid, evaporating the solution to dryness, and extracting the residue with alcohol to remove traces of ammonium chloride. It crystallises from alcoholic ether, melts at 234—236° with decomposition, and is readily soluble in water and alcohol, but insoluble in ether. The *platinochloride*, $(C_5H_{11}N)_2,H_2PtCl_6$, is crystalline. The free *base* is an oil boiling at 82—83°; it has a strongly basic odour, an alkaline reaction, and absorbs carbonic anhydride from the air.

Tetramethylenylcarbamide, $C_4H_7{\cdot}CH_2{\cdot}NH{\cdot}CO{\cdot}NH_2$, prepared by evaporating a solution of the hydrochloride of the amine with potassium cyanate, crystallises from hot alcohol in long, scaly needles, melts

at 116°, and is readily soluble in hot, but only sparingly in cold water.

Tetramethylenylthiocarbamide, $C_4H_7\cdot CH_2\cdot NH\cdot CS\cdot NH_2$, prepared by boiling the hydrochloride of the amine with ammonium thiocyanate, crystallises from water or alcohol in slender needles and melts at 67—68°. F. S. K.

Tetric Acid and its Homologues. By R. Moscheles and H. Cornelius (*Ber.*, **21**, 2603—2609).—*Ethyl tetrate*, $C_7H_{10}O_3$, forms lustrous crystals, melts at 30°, and boils at 180° under 70 mm. pressure. It dissolves in boiling aqueous soda, and is precipitated unchanged by dilute sulphuric acid. The ethyl salts of pentic and phenyltetric acids are oils. None of these salts have any analogy with ethyl succinosuccinate: they are colourless, dissolve sparingly in water, readily in alcohol and ether; the solutions show no fluorescence; they are readily hydrolysed by hydrogen chloride and bromide, but not by alkalis. It is therefore improbable that tetric acid and its homologues are alkylated succinosuccinic acids (Wedel, *Annalen*, **219**, 106). The formation of tetric acid from ethyl bromomethylacetoacetate is best explained by the formula $<^{CHMe\cdot CO}_{CO\cdot CH_2-}>O$ (Michael, *J. pr. Chem.* [2], **37**, 503). It is probable that in the formation of alkyl salts an intramolecular migration takes place, the group $=CH\cdot CO-$ becoming $=C(OH)\cdot C=$, as the salts do not react with phenylhydrazine, whilst the acids do.

Pentic acid phenylhydrazide, $C_6H_8O_2\cdot N_2HPh$, prepared by dissolving pentic acid (1 mol.) and phenylhydrazine (1 mol.) in 50 per cent. acetic acid, is crystalline.

Tetrinamide, $C_5H_7NO_2$, obtained by heating the acid with alcoholic ammonia at 150°, melts at 212°.

Bromotetric acid, $C_5H_5O_3Br$, forms white crystals melting at 75°.

Phenyltetric acid, $C_{11}H_{10}O_3$, is prepared by heating ethyl bromobenzylacetoacetate in a water-bath. It is sparingly soluble in hot water, and is much more stable than the other acids of the group. The *benzoyl-derivative*, $C_{18}H_{14}O_4$, crystallises from alcohol in needles melting at 110°. N. H. M.

Constitution of Mesitonic Acid. By R. Anschütz and C. Gillet (*Annalen*, **247**, 99—111).—The authors regard mesitonic acid as *α-dimethyllevulinic acid*, as it closely resembles levulinic acid in its behaviour with certain reagents. A crystalline compound is deposited when phenylhydrazine acetate is added to an aqueous solution of mesitonic acid. The compound melts at 121·5° and forms prismatic crystals soluble in benzene and alcohol. It is very unstable and is decomposed by boiling with hydrochloric acid, yielding the compound $CMe_2<^{CH_2\cdot CMe}_{CO\cdot NPh-}>N$. Mesitonic acid yields dimethylmalonic acid when oxidised with nitric acid, and α-dimethylvalerolactone when reduced with a large excess of sodium amalgam.

α-Dimethylvalerolactone is crystalline, melts at 52°, and boils at 86° under 15 mm. pressure. In the formation of dimethyllevulinic acid from mesityl oxide, $CMe_2{:}CH\cdot COMe$, a hydrochloric acid additive pro-

duct, $CMe_2Cl\cdot CH_2\cdot COMe$, is first formed; this is converted into the nitrile of α-dimethyllevulinic acid, $CN\cdot CMe_2\cdot CH_2\cdot COMe$, by the action of potassium cyanide, and on saponification the amide and the free acid are produced. W. C. W.

Ethyl Oxallevulinate. By W. Wislicenus (*Ber.*, **21**, 2583—2586).—*Ethyl oxallevulinate*, $C_{11}H_{16}O_6$, is prepared by adding rather more than 1 mol. of ethyl oxalate to sodium ethoxide previously treated with 10 parts of absolute ether; after 12 hours, the calculated amount of ethyl levulinate is added. The whole is left for some days, cooled and shaken with water; the aqueous solution is separated from the ether, and made acid, when the ethyl oxallevulinate separates as a light oil. It is purified by means of the copper or potassium compound and can then be distilled. It is a thick, colourless oil, boils at 198° under 27 mm. pressure, is insoluble in water, readily soluble in alcohol and ether. The alcoholic solution gives an intense red colour with ferric chloride. The *copper compound*, $(C_{11}H_{15}O_6)_2Cu$, crystallises from much boiling absolute alcohol in very small, dark green needles which melt with decomposition at 190—200°. It is insoluble in water. The *potassium compound* crystallises in hair-like, matted, lustrous needles, melts at 180—183° with evolution of gas, and is very readily soluble in water.

When ethyl oxallevulinate is heated with phenylhydrazine on a water-bath, the compound $C_{17}H_{20}N_2O$ is obtained. This crystallises in groups of lustrous needles, melts at 83—84°, and is readily soluble in ether, benzene, and hot alcohol. It is a feeble base, and dissolves in strong hydrochloric acid, but is precipitated on diluting. When boiled with alcoholic potash, the potassium salt of the acid $C_{13}H_{12}O_4N_2$ is formed. The *free acid* crystallises in needles with 1 mol. H_2O, melts at 165—167°, and dissolves readily in alcohol and in aqueous sodium carbonate. N. H. M.

Pimelic Acid from Menthol. By G. Arth (*Compt. rend.*, **107**, 107—110).—The acid, $C_7H_{12}O_4$, produced by the oxidation of menthol (*Ann. Chim. Phys.* [6], **7**, 439), is not identical with normal propylsuccinic acid prepared by Waltz's method. The amide of the former is much more soluble in water and crystallises in prismatic crystals melting at 191°. The amide of the latter separates in small, hard crystals or in opaque, dendritic groups. C. H. B.

Determination of the Molecular Weight of Dimethyl Diacetylracemate by Raoult's Method. By R. Anschütz (*Annalen*, **247**, 111—122).—Dimethyl diacetylracemate is formed when a benzene solution of the dimethyl salts of the dextro- and lævo-gyrate diacetyltartaric acids evaporate spontaneously. The crystals melt at 86°, and are identical in every respect with the compound obtained by boiling dimethylracemate with acetic chloride.

Determinations of the molecular weight by Raoult's method gave unexpected results. The molecular weight for dimethyl diacetyldextrotartrate and of dimethyl diacetylracemate are identical, whilst it

was to be expected that the latter would have had a molecular weight double that of the former. W. C. W.

Water of Crystallisation of certain Pyrotritartrates. By C. Böttinger (*Annalen*, **274**, 255—256).—In a former paper, the author stated that the barium and calcium salts of pyrotritartaric (*Annalen*, **172**, 242) (nvic) acid are deposited from dilute solutions with 5 and 6 mols. H_2O respectively:—$(C_7H_7O_3)_2Ba + 5H_2O$ and $(C_7H_7O_3)_2Ca + 6H_2O$. From concentrated solutions, the salts crystallise with 4 mols. H_2O, *e.g.*, $(C_7H_7O_3)_2Ba + 4H_2O$. W. C. W.

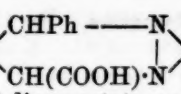
Action of Ethereal Salts of Diazoacetic Acid on Ethereal Salts of Unsaturated Acids. By E. Buchner (*Ber.*, **21**, 2637—2647).—*Methyl fumaricdiazoacetate*, $\begin{matrix}CH(COOMe)\cdot N\\ \\ CH(COOMe)\cdot N\end{matrix}\!\!>\!CH\cdot COOMe$, is obtained when methyl fumarate (1 mol.) and methyl diazoacetate (1 mol.) are warmed together. The reaction commences at about 60°, and a considerable amount of heat is generated. The product, which was only obtained in an impure condition, is a thick, yellowish oil, and is readily soluble in hot water, but is not miscible with ether in all proportions. It immediately decolorises alkaline potassium permanganate, and dissolves in aqueous or alcoholic potash with a transient, but intense, yellow coloration, forming a colourless *potassium* salt, from which the *acid* was obtained in the form of a yellowish, crystalline mass, melting at 220° with decomposition. The *barium* salt is readily soluble. The methyl salt is decomposed when boiled with mineral acids, yielding carbonic anhydride, the corresponding hydrazine salt, and an acid, probably succinic acid.

Trimethyl 1.2.3-*trimethylenetricarboxylate*, $C_9H_{12}O_6$, is obtained when the preceding compound is heated; at about 160°, evolution of gas commences, reaching its maximum at about 200°; on continued heating, a colourless liquid distils, which solidifies to a crystalline mass on cooling. It crystallises from boiling alcohol or water in colourless needles, melts at 61°, boils at 267° (732 mm.), and is readily soluble in alcohol, ether, benzene, and light petroleum. It is not acted on by bromine or by alkaline potassium permanganate.

Trimethylenetricarboxylic acid is obtained when the methyl salt is hydrolysed with alcoholic potash (compare Perkin, Trans., 1885, 801). It crystallises from ether in small, colourless needles, melts at 220°, and is sparingly soluble in ether. The ammonium salt crystallises in plates, and is readily soluble. The *anhydride*,

$$COOH\cdot C_6H_3\!<\!\begin{matrix}CO\\CO\end{matrix}\!>\!O,$$

is formed when the acid is distilled in a partial vacuum. It crystallises from ether in small prisms, melts at 186—189°, boils at 265° (75 mm.), is readily soluble in water and alcohol, and moderately soluble in ether. The *lead*, *barium*, and *silver* salts of the anhydride are moderately soluble in water, the *calcium* salt readily.

Ethyl cinnamicdiazoacetate,  , prepared by heating a mixture of ethyl diazoacetate and ethyl cinnamate to 110°, and then very gradually to 130°, crystallises from alcohol in colourless prisms, melts at 79°, and is readily soluble in alcohol, ether, benzene, and glacial acetic acid. It dissolves in alkalis with a transient, yellow coloration, is not acted on by cold concentrated sulphuric acid, and is decomposed into nitrogen and ethyl phenyltrimethylenedicarboxylate when heated above its melting point.

Cinnamicdiazoacetic acid, $C_{11}H_{10}N_2O_4$, prepared by hydrolysing the ethyl salt with soda, separates from water in almost colourless, nodular crystals, melts at 178° with decomposition, is readily soluble in boiling water, and moderately soluble in alcohol and ether. It is completely decomposed when heated at 130° for some time, but is only slowly acted on by cold, alkaline potassium permanganate. It yields a colourless bromo-derivative, which crystallises from water and melts at 227° with decomposition. When reduced with zinc and glacial acetic acid, it gives an acid which crystallises in needles and is readily soluble in water. The *silver* salt, $C_{11}H_8N_2O_4Ag_2$, is insoluble in water, and when heated at 140° gives a nitrogenous compound; this sublimes in needles, and crystallises from alcohol in plates melting at 228°. The *barium* salt is readily soluble in water.

Ethyl phenyltrimethylenedicarboxylate, $C_{15}H_{18}O_4$, is obtained when ethyl cinnamicdiazoacetate is distilled under diminished pressure. It is a thick oil, boils at 256—257° (120 mm.), and is only slowly oxidised by alkaline potassium permanganate. It is not acted on by bromine in cold acetic acid solution.

Phenyltrimethylenedicarboxylic acid, $C_{11}H_{10}O_4$, crystallises from alcohol in colourless prisms, melts at 175°, is readily soluble in alcohol and ether, and moderately in water. It is not oxidised by alkaline potassium permanganate. An acid, probably phenyltrimethylenecarboxylic acid, is obtained when the dicarboxylic acid is distilled under diminished pressure. It separates from ether in colourless crystals, melts at 134°, boils at 282° (190 mm.), and is not oxidised by alkaline potassium permanganate. The *ammonium* salt is crystalline; the *silver* salt is sparingly soluble in water. Ethyl orthonitrophenylpropiolate combines with ethereal salts of diazoacetic acid, but the author has not hitherto succeeded in isolating the intermediate nitrogenous product. F. S. K.

Action of Sulphuric Acid on Bromo-derivatives of Benzene. By J. Herzig (*Monatsh.*, **9**, 586—588).—The author has confirmed his earlier experiments (Abstr., 1882, 46), and refuses to accept the explanation of them offered by Jacobsen (this vol., p. 137). In conclusion he points out the very different behaviour of monobromodurene and monobromobenzene towards sulphuric acid, the reaction in the former case being comparatively simple, and admitting of ready explanation, whilst the latter is only attacked with difficulty and at relatively high temperatures, furnishing compounds not nearly so intimately related to bromobenzene. G. T. M.

Tribromotrinitrobenzene. By C. L. JACKSON and J. F. WING (*Amer. Chem. J.*, **10**, 283—294).—Koerner has stated that he has prepared tribromotrinitrobenzene, but Wurster and Bevan have denied it. The authors, however, have prepared this substance by Koerner's method, and shown that his critics failed by using a nitric acid containing nitrous acid (compare this vol., p. 136).

Tribromotrinitrobenzene, $C_6Br_3(NO_2)_3$.—Symmetrical tribromobenzene is first converted into tribromodinitrobenzene, and this then further nitrated by a mixture of nitric acid of sp. gr. 1·51 and fuming sulphuric acid; the product requires purifying from unaltered material and from tetrabromodinitrobenzene (see below). The yield is from 15 to 20 per cent. of the theoretical. It crystallises well from chloroform or benzene, is almost insoluble in alcohol, and melts at 285° (uncorr.). It reacts readily with a number of substances, but few of the products have been investigated. When treated with alcoholic ammonia, a pale-yellow, amorphous substance separates, namely, *triamidotrinitrobenzene*, $C_6(NH_2)_3(NO_2)_3$. It is insoluble in ordinary reagents, but may be crystallised from aniline or nitrobenzene; it does not react with acids. With reducing agents, it should yield hexamidobenzene, but the reduction, apparently best effected with zinc-dust and acetic acid, is attended with many difficulties.

Trianilidotrinitrobenzene, $C_6(NHPh)_3(NO_2)_3$, is obtained by allowing aniline to act on tribromotrinitrobenzene in the cold. It is an orange powder, insoluble in water, but soluble in benzene, &c., and melts at 238°. It has no basic properties.

Tetrabromodinitrobenzene, $C_6Br_4(NO_2)_2$, is formed as above mentioned in the preparation of tribromotrinitrobenzene, and in about equal quantity. Its separation is tedious, and is effected by fractional crystallisation from a mixture of alcohol and benzene. It melts at 224°, whereas Richter found the melting point to be 227—228°. The way in which it is formed is not understood. H. B.

Electrolysis of Phenol by Alternating Currents. By E. DRECHSEL (*J. pr. Chem.* [2], **38**, 65—74).—By distilling the liquid resulting from his former experiments (Abstr., 1884, 1136), the author obtained an aqueous liquid containing red, oily drops. These were dissolved in ether, and the solution fractionated; a liquid boiling between 153° and 178° was thus obtained, which on analysis gave the formula $C_6H_{10}O$. By treating the oil with phenylhydrazine, a crystalline substance was produced; this was distilled with dilute sulphuric acid to try and obtain the above-mentioned liquid, but very little distilled over, and most of it was left in the form of black, oily drops floating on the liquid in the retort. These drops crystallised on cooling, and after recrystallisation from alcohol gave rhomboidal tables which rapidly became brown when exposed to the light. A purer product was obtained from the mother-liquor, and was further purified by sublimation in a vacuum and subsequent recrystallisation from 50 per cent. alcohol. The crystals were brilliant, white lamellæ, slightly soluble in hot water and freely soluble in alcohol; they melt at 108°, and have the formula $C_{12}H_{13}N$. This substance is isomeric with Graebe and Glaser's hydrocarbazol, which melts at 120°.

The author proposes the name *hydrophenoketone* for the substance $C_6H_{10}O$, which is isomeric with mesityl oxide, and *hydrophenanilide* for the compound $C_{12}H_{13}N$.

The author has previously noticed the formation of caproic acid by the action of the alternating current on phenol; he now explains this by supposing that hydrophenoketone is an intermediate product, produced by the alternate hydrogenation and oxidation of the phenol. This conversion of substances of the aromatic series into those of the fatty series may serve to throw light on the changes that take place among these and allied substances in the animal body.

A. G. B.

Action of Chlorine on Catechol and Orthamidophenol. By T. Zincke and F. Küster (*Ber.*, **21**, 2719—2730).—*Hexachlorodiketotetrahydrobenzene*, $CCl\ll^{CCl\cdot CCl_2}_{CCl_2\cdot CO}>CO + H_2O$, can be prepared by dissolving catechol in 20 parts of glacial acetic acid and saturating the solution with chlorine; the mixture is kept cold at first, but as soon as tetrachlorocatechol begins to separate, the cooling is not necessary. After keeping for one or two days, the solution is poured into water, the precipitate collected, and the solution extracted with ether. It can also be prepared by passing chlorine through a well-cooled glacial acetic acid (10 parts) solution of orthamidophenol hydrochloride until the crystals of tetrachloramidophenol which separate are redissolved; on evaporating, the product separates, and is purified by washing with water. The yield by this method is as much as 80 per cent. of the theoretical quantity. It separates from a mixture of ether and light petroleum in colourless or slightly yellow crystals which contain 2 mols. H_2O, but lose 1 mol. H_2O when kept in a partial vacuum or when recrystallised from hot, light petroleum. The monohydrate is stable, but loses its water when heated at 90—95° for a long time. It is readily soluble in alcohol, glacial acetic acid, benzene, and ether, and melts at 93—94° with decomposition. When treated with aniline, it yields a small quantity of a compound melting at 236—237°. It liberates iodine from potassium iodide, and when reduced with stannous chloride it yields tetrachlorocatechol.

Hexachloropentenehydroxycarboxylic acid, $\ll^{CCl\cdot CCl_2}_{CCl\cdot CCl_2}>C(OH)\cdot COOH$, prepared by dissolving the preceding compound in sodium acetate or sodium carbonate and adding hydrochloric acid to the filtered solution, crystallises from dilute acetic acid with 1 mol. H_2O, and from light petroleum in small, colourless prisms. It melts at 111°, and on continued heating quickly solidifies at 140—150°, melting again at 185—186°. When the aqueous solution is heated, yellow, scaly crystals separate; this compound melts at 153°, and yields a red anilide. The *barium* salt, prepared by neutralising an alcoholic solution of the acid with barium hydrate, crystallises in needles which appear to contain alcohol (1 mol.); it is decomposed when heated at about 50°. The *methyl* salt, $OH\cdot C_5Cl_6\cdot COOMe$, prepared by boiling hexachlorodiketotetrahydrobenzene with methyl alcohol, crystallises from light petro-

leum in colourless needles, melts at 62°, and is readily soluble in methyl and ethyl alcohol. The *ethyl* salt, obtained in like manner, crystallises from alcohol in rhombic prisms, and from hot, light petroleum in white needles. It melts at 121°, and dissolves unchanged in soda, but is insoluble in sodium carbonate. The *acetyl*-derivative melts at 85°, and is readily soluble in ordinary solvents, but insoluble in dilute soda.

Hexachloroketopentene, $\begin{matrix}CCl\cdot CCl_2\\CCl\cdot CCl_2\end{matrix}\!\!>\!CO$, is obtained when the preceding compound is dissolved in glacial acetic acid, a little hydrochloric acid added, and the solution warmed with a slight excess of a 10 per cent. chromic acid solution. It crystallises in colourless, rhombic plates, melts at 31·5°, boils at 155—158° (80 mm.), and can be distilled with steam. It is insoluble in sodium carbonate. The *anilide*, $C_5Cl_5(NHPh)O$, crystallises in small, yellow needles or plates melting at 194—196°.

Pentachlorcbutenecarboxylic acid, $C_5Cl_5O_2H$, prepared by dissolving the preceding compound in 10 per cent. soda, crystallises from hot, dilute acetic acid in needles or prisms, melts at 127°, and is readily soluble in ether, alcohol, and benzene, but only sparingly in light petroleum. It distils with partial decomposition, and loses hydrogen chloride when heated with concentrated sulphuric acid. When reduced with sodium amalgam, it yields a liquid acid, which has a strong, fatty-acid-like odour, and forms a readily soluble barium salt. The *ammonium* salt crystallises in white needles, melting at 178—180° with decomposition. The *barium* salt is readily soluble, but appears to be unstable in aqueous solution. The *methyl* and *ethyl* salts are liquids. The constitution of the acid is probably $CCl_2{:}C{:}CCl\cdot CCl_2\cdot COOH$.

Tetrachlorocatechol, $C_6Cl_4(OH)_2$, is obtained when hexachlorodiketohydrobenzene is reduced with stannous chloride in glacial acetic acid solution. It crystallises from hot dilute alcohol or benzene in slender needles, melts at 194—195°, and, when oxidised with nitric acid, yields tetrachlororthobenzoquinone, melting at 129—130° (compare Abstr., 1887, 808). The *acetyl*-derivative crystallises from acetic acid in thick needles melting at 190°. F. S. K.

Iodine-derivative of Quinone. By K. Metzeler (*Ber.*, **21**, 2554—2556).—*Diiodoquinone*, $C_6H_2I_2O_2$, is formed when diacetylquinol (1 part) is slightly warmed on a water-bath with a solution of potassium iodate (4 parts), slightly acidified with sulphuric acid. It crystallises from alcohol in yellow needles melting at 157—159°. When suspended in water and treated with sulphurous acid, *diiodoquinol* is obtained. This crystallises in slender needles of a silky lustre, melts at 142·5° (uncorr.), and dissolves readily in hot water, alcohol, ether, and glacial acetic acid. *Diacetyldiiodoquinol* crystallises in rhombic plates and in slender needles, melts at 148° (uncorr.), is insoluble in water, readily soluble in alcohol, ether, and in hot benzene and glacial acetic acid. N. H. M.

Some New Benzene-derivatives. By M. E. G. Körner and V. Wender (*Gazzetta*, **17**, 486—494).—The authors point out how

necessary it is for the advancement of our knowledge of the chemico-physical properties and structure of the benzene-derivatives, that the various gaps in the different series should be filled, and the properties of the compounds which fill these gaps compared with those of the other analogous derivatives of the series. With this object, they have prepared several new benzene-derivatives, and at the end of the paper give in tabular form the chief chemico-physical properties of these various ortho-, meta-, and para-compounds.

Orthiodaniline, $C_6H_4I{\cdot}NH_2$.—When orthiodonitrobenzene (m. p. 49·5°) is acted on by the ordinary reducing agents, it is decomposed with elimination of the iodine, but this is not the case if ferrous hydroxide is employed; then the NO_2 group alone is attacked and converted into the NH_2 group. The iodonitrobenze was obtained by the nitration of iodobenzene and also from orthonitraniline. A small quantity only of the nitro-compound should be operated on at a time (25 grams), using ferrous sulphate (250 grams) and ammonia in slight excess, diluted with its own volume of water. The mixture is heated on a water-bath for 8 to 10 hours, and when cold, the iodaniline is extracted with ether, and distilled in a current of steam. It is then dissolved in dilute sulphuric acid, agitated with ether, to remove unaltered nitro-compounds, the solution precipitated with ammonia or an alkaline carbonate, and the iodaniline redistilled in steam. The yield is more than two-thirds of the theoretical.

Orthiodaniline has a disagreeable odour, somewhat resembling that of the pyridine bases; it is less soluble in water than pariodaniline, the boiling solution as it cools depositing the substance in long, flexible needles of silky lustre; in other solvents it dissolves freely; exposed to air and light, it becomes brown after a time. It is much more volatile than its isomerides, and passes over easily with the vapour of water. It melts at 56·5°, and if heated sharply decomposes with evolution of iodine vapour.

When acted on with iodine, it yields Rudolph's diiodaniline, melting at 96°; this confirms the constitution 1 : 2 : 4 for this diiodaniline. The salts of orthiodaniline are more soluble in water, but less stable than those of the corresponding isomerides, decomposing in aqueous solution unless there is a large excess of acid present. The *hydrochloride*, $C_6H_4I{\cdot}NH_2,HCl + H_2O$, forms short prisms; the *platinochloride* is an orange-yellow, crystalline powder. The *sulphate*, $3C_6H_4I{\cdot}NH_2,2H_2SO_4$, separates from water in silky needles grouped in nodules; from alcohol in brilliant plates.

For comparison, the authors also prepared the two known iodanilines; *pariodaniline* melts at 63°; *metaiodaniline* at 27°; the hydrochloride of the latter crystallises in prisms.

Orthiodacetanilide, $C_6H_4I{\cdot}NHAc$ = [1 : 2].—This is easily prepared by gently boiling a mixture of orthiodaniline and acetic anhydride in molecular proportion for four or five hours. Purified by crystallisation from alcohol, with use of animal charcoal, it forms slender prisms or large, hexagonal tables. It melts at 109·5—110°, and is much more volatile than its isomerides, subliming readily when gently heated.

Metaiodacetanilide, $C_6H_4I{\cdot}NHAc$ = [1 : 3].—This, obtained like

the ortho-derivative, is more soluble than the latter, and crystallises in brilliant needles. It melts at 119·5°, and can be sublimed.

Pariodacetanilide, $C_6H_4I{\cdot}NHAc$ = [1 : 4].—This is less soluble than its isomerides. It crystallises from alcohol in rhombic prisms, which may be obtained of large size by spontaneous evaporation. It melts at 183°, and sublimes, but with difficulty.

Orthodiiodobenzene, $C_6H_4I_2$.—This is prepared by dissolving the iodaniline (10 grams) in hot dilute sulphuric acid (9 grams acid to 70 c.c. of water), cooling to 0° with continual agitation, adding a solution of potassium nitrite (4·5 grams) in a little water, and then excess of solution of potassium iodide in hydriodic acid of sp. gr. 1·1. After leaving the mixture for several hours at the ordinary temperature, the dark-coloured oil which separates is collected and distilled with steam in the presence of a little potash. Orthodiiodobenzene is a colourless liquid, which solidifies to a crystalline mass when cooled. It melts at 27°, and boils at 286·5° under a pressure of 751·5 mm. Its odour resembles that of its isomerides. *Nitro-orthodiiodobenzene*, $C_6H_2I_2{\cdot}NO_2$ = [1 : 2 : 4 probably]. This is prepared by dissolving the diiodobenzene in nitric acid of sp. gr. 1·54, and after a few minutes diluting with water. When purified by crystallisation from alcohol, it forms small, lemon-yellow prisms which melt at 112·5°.

Orthanisidine, $NH_2{\cdot}C_6H_4{\cdot}OMe$ = [1 : 2] is known. The authors found the boiling point to be 218° under a pressure of 755·5 mm.

Metanisidine was prepared by the reduction of metanitransoïl with tin and hydrochloric acid. It remains liquid at −12°, and boils at 243·5° under 755·5 mm. pressure. *Paranisidine* boils at 239·5° at 755 mm.

Orthacetaniside, $NHAc{\cdot}C_6H_4{\cdot}OMe$, prepared by boiling for some hours a mixture of orthanisidine with acetic anhydride in molecula proportion, crystallises in hexagonal tables, and melts at 84°. *Metacet aniside* melts at 80—81°, and crystallises from water in needles or scales, and from alcohol in flat tables or elongated octahedra *Paracetaniside* forms hexagonal tables, which melt at 127·1°

C. E. G.

Action of Aniline on Epichlorhydrin. By A. Fauconnier (*Compt. rend.*, **107**, 250—252).—The base, $C_{15}H_{16}N_2O$, obtained by the action of aniline on epichlorhydrin (this vol., p. 586), crystallises from boiling alcohol in long, slender, white needles, which melt at 53—54°, and rapidly become violet in colour when exposed to light even in a closed vessel.

The *oxalate*, $(C_{15}H_{18}N_2O,H_2C_2O_4)_2 + 3H_2O$, separates in bundles of slender needles when an ethereal solution of the base is mixed with an aqueous solution of the acid. When purified by washing with water and recrystallisation from alcohol, it melts at 149—150°, and slowly becomes lemon-yellow when exposed to light.

The *platinochloride*, $C_{15}H_{18}N_2O,H_2PtCl_6 + 4H_2O$, crystallises in small, brilliant yellow lamellæ.

The *acetyl-derivative*, $C_{15}H_{17}N_2OAc + H_2O$, is obtained by heating the base for several hours with three times its weight of acetic anhydride and crystallising the product from alcohol. It forms colourless

lamellæ, which seem to be hexagonal, but which when dried become opaque and break up into prismatic crystals melting at 99—100°.

A *nitroso-derivative*, $C_{15}H_{16}N_2O(NO)_2$, is precipitated as a resin when aqueous solutions of the hydrochloride and sodium nitrite are mixed together. It crystallises from boiling alcohol in orange needles, which melt at 108—109°. Attempts to convert it into a hydrazine-derivative were unsuccessful.

It is evident from the formation of these compounds that the base contains one hydroxyl-group, and has a double basic function. The author prefers to call it *hydroxypropylenediphenyldiamine*, and not dianilglycerol as at first proposed (*loc. cit.*). C. H. B.

Action of Aniline on Bromofumarimide. By K. Löscher and R. Kusseroff (*Ber.*, **21**, 2718—2719).—When bromofumarimide is heated with excess of aniline, a compound is obtained which has the composition of *anilidofumarimide*, and seems to have the constitution $\ll^{C(NHPh)-}_{CH\cdot CO\cdot NH}>CO$. It crystallises from hot dilute alcohol in golden-yellow plates, melts at 202°, and is readily soluble in glacial acetic acid and boiling alcohol, sparingly soluble in hot water, ether, chloroform and benzene, and insoluble in cold water. It yields an *acetyl*-derivative, and when heated with alcoholic ammonia, a crystalline compound, probably anilidofumaramide, is obtained. F. S. K.

Compounds of Organic Bases with Salts of the Heavy Metals. By B. Lachowicz and F. Bandrowski (*Monatsh.*, **9**, 510—517).—Zinc chloride forms compounds of the general formula $x(B) + yZnCl_2 + 2ZnO$, with all fatty compounds having a decidedly basic character, such as primary, secondary, and tertiary amines, neurine, and guanidine. In the aromatic series, it combines only with those primary bases which contain one benzene nucleus, or such as piperidine, pyridine, and picoline, but not with such compounds as naphthylamine.

When aniline is shaken with an aqueous solution of zinc chloride, a white, crystalline precipitate of the formula $2NH_2Ph,ZnCl_2 + 2H_2O$ is formed. From hot alcohol it crystallises in long needles, which do not decompose when heated to 230°. With substituted anilines containing the negative groups Cl, NO_2, and OH, zinc chloride does not combine, but with orthotoluidine it gives the compound

$$2C_7H_7\cdot NH_2,ZnCl_2 + 2H_2O,$$

with paratoluidine, $2C_7H_7NH_2,ZnCl_2 + 3H_2O$, and with metaxylidine, $2C_8H_9NH_2,ZnCl_2$.

α-Naphthylamine does not combine either with copper sulphate or with mercuric chloride, but with mercurous nitrate it forms a characteristic compound. β-Naphthylamine differs from its isomeride in combining with both mercuric chloride and copper sulphate.

The authors suggest that the property of combining with certain metallic salts may furnish a convenient method of isolating many organic bases. G. T. M.

Some Anhydro-bases from Unsymmetrical Metaxylidine. By E. GUDEMAN (*Ber.*, **21**, 2549—2553).—*Thioformo-xylide*,

$$C_6H_3Me_2\cdot NH\cdot CSH,$$

is obtained by heating 25 grams of formo-xylide (Abstr., 1885, 772) with 16 grams of phosphorus pentasulphide for 30 minutes on a water-bath, extracting the resinous product with dilute aqueous potash, and precipitating with hydrochloric acid. It crystallises from alcohol in small, slender needles melting at 105°.

Methenylamido-xylil mercaptan, $C_6H_2Me_2<\begin{matrix}N\\S\end{matrix}>CH$, is formed by treating thioformo-xylide with potassium ferricyanide in alkaline solution (Jacobsen, Abstr., 1886, 700). It is a heavy, yellowish liquid, of a repulsive odour.

Thioaceto-xylide, $C_6H_3Me_2\cdot NH\cdot CSMe$, is prepared by heating 10 grams of aceto-xylide (m. p. 127°) for half an hour with 6 grams of phosphorus pentasulphide on a water-bath. It melts at 80°.

Ethenylamido-xylil mercaptan, $C_6H_2Me_2<\begin{matrix}N\\S\end{matrix}>CMe$, obtained by adding 10 grams of thioaceto-xylide in 300 c.c. of water with 75 c.c. of 33 per cent. aqueous soda to 60 grams of potassium ferricyanide in 200 c.c. of water, is an oil.

Thiobenzo-xylide, $C_6H_3Me_2\cdot NH\cdot CSPh$, is prepared by heating 50 grams of benzo-xylide with 35 grams of phosphorus pentasulphide on a sand-bath; it crystallises from alcohol in small needles, which melt at 90°.

Benzo-xylide, $C_6H_3Me_2\cdot NHBz$, obtained by heating 24 grams of xylidine with 36 grams of benzoic acid for eight hours at 200—230°, and then distilling, crystallises from alcohol in long needles, which melt at 187—189°.

Benzenylamido-xylil mercaptan, $C_6H_2Me_2<\begin{matrix}N\\S\end{matrix}>CPh$, obtained by oxidising thiobenzo-xylide with potassium ferricyanide, forms a white oil, which becomes greenish-yellow when exposed to air.

Ortho- and *para-benzotoluide* and *benzocumide* are readily obtained by heating the amines with an excess of benzoic acid; they are readily soluble in alcohol and in strong hydrochloric acid, insoluble in water, and melt at 131°, 154°, and 167° respectively.

N. H. M.

Dimethylaniline and Diphenylamine Sulphates. By L. VIGNON (*Compt. rend.*, **107**, 263—266).—Anhydrous dimethylaniline and sulphuric acid combine at the ordinary temperature with development of heat and formation of a crystalline acid sulphate, NMe_2Ph,H_2SO_4, which can be purified by washing with benzene, and melts at 80°. It dissolves in water, but is insoluble in ether and in benzene. No normal sulphate is formed whatever the proportions in which the acid and base are mixed.

If the acid sulphate is heated at 180—190°, it loses water and is converted into the sulphonic acid, $C_6H_4(SO_3H)\cdot NMe_2$, the barium salt of which crystallises with both 3 mols. and 5 mols. H_2O.

Diphenylamine, previously fused, likewise combines with sulphuric

acid, forming a crystalline acid sulphate whatever the proportions in which they are mixed. It has the composition $NHPh_2,H_2SO_4$, and after purification by washing with benzene melts at 123—125°. The salt is insoluble in ether and benzene, and when thrown into water is decomposed, the diphenylamine separating as a flocculent precipitate.

When heated at 180—190° for two hours, the acid sulphate loses water and is converted into the sulphonic acid, $C_6H_4(SO_3H)\cdot NHPh$, the barium salt of which forms anhydrous crystals.

The heats of formation of the solid salts and of aniline sulphate from the bases and dilute sulphuric acid were determined by treatment with soda with the following results:—

Aniline sulphate	16·6 Cals.
Dimethylaniline sulphate.....	8·4 ,,
Diphenylamine sulphate.....	4·5 ,,

The conversion of aniline sulphate into sulphanilic acid by the action of heat is well known, and it would seem that this reaction is general. The formation of sulphonic acids is generally attributed to the dehydrating action of the excess of sulphuric acid which is always employed; but this explanation will not hold in the three cases just described. In these instances, the change is due to interatomic forces which become active at a high temperature. C. H. B.

Silicotetrafluorides of Certain Bases. By A. M. Comey and F. W. Smith (*Amer. Chem. J.*, **10**, 294—296: compare this vol., p. 941).—*Trinitrosodimethylaniline disilicotetrafluoride*,

$$(C_8H_{10}N_2O_3)_3(SiF_4)_2,$$

is formed by passing silicon fluoride into a solution of the base in benzene; it is bright yellow and amorphous, and decomposes completely at 150°. *Dipyridine silicotetrafluoride*, $(C_5NH_5)_2SiF_4$, is prepared in like manner; it is white, amorphous, and decomposes after a time. *Tripyridine disilicotetrafluoride*, $(C_5NH_5)_3(SiF_4)_2$, is obtained by heating the preceding substance, when pyridine is liberated, and the new compound sublimes in crystalline crusts. H. B.

Diazoamido-compounds. By H. Goldschmidt and E. Molinari (*Ber.*, **21**, 2557—2579).—*Diazobenzenediphenylcarbamide*,

$$N_2Ph\cdot NPh\cdot CO\cdot NHPh,$$

is obtained by slightly heating a mixture of diazobenzene with phenyl cyanate in molecular proportion, dissolved in light petroleum; on cooling, it separates as an almost white, crystalline precipitate, which melts at 125°. When mixed with 10 per cent. sulphuric acid and steam-distilled, it is decomposed into phenol, phenyl cyanate, and diphenylcarbamide, the latter being partially decomposed into aniline and carbonic anhydride.

Diazobenzenephenylparatolylcarbamide, $C_7H_7\cdot NH\cdot CO\cdot NPh\cdot N_2Ph$, prepared in a manner similar to the diphenylcarbamide, crystallises in slender, microscopic needles melting at 134°. It is decomposed by acids into paratolylphenylcarbamide, aniline, paratoluidine, ditolylcarbamide, and phenol.

Paradiazotolueneparatolylphenylcarbamide,

$$NHPh\cdot CO\cdot N(C_7H_7)\cdot N_2\cdot C_7H_7,$$

forms slender, white needles, melts at 116°, and is decomposed by acid with formation of paratolylphenylcarbamide, aniline, toluidine, diphenylcarbamide, and paracresol.

Paradiazotolueneparaditolylcarbamide, $C_7H_7\cdot NH\cdot CO\cdot N(C_7H_7)\cdot N_2\cdot C_7H_7$, crystallises in long, white needles melting at 140°.

Diazobenzeneparatolylphenylcarbamide, $NHPh\cdot CO\cdot N(C_7H_7)\cdot N_2Ph$, is formed when an ethereal solution of diazoamidobenzenetoluene (from diazobenzene chloride and toluidine) is warmed with phenyl cyanate; it is a white precipitate consisting of slender needles, and may be purified by extraction with boiling ether; it melts at 126°. When decomposed with sulphuric acid, paratolylphenylcarbamide, as well as aniline, paratoluidine, phenol, paracresol, and phenyl cyanate, are formed. This reaction establishes the constitution of the carbamide as given, and also that of the diazoamidobenzenetoluene, which is $N_2Ph\cdot NH\cdot C_7H_7$.

Diazobenzeneparaditolylcarbamide, $C_7H_7\cdot NH\cdot CO\cdot N(C_7H_7)\cdot N_2Ph$, prepared from diazoamidobenzenetoluene and paratolyl cyanate, forms small, thick, lustrous prisms melting at 130°. It is decomposed by sulphuric acid into paraditolylcarbamide, aniline, paratoluidine, phenol, and paracresol.

Diazobenzene-β-naphthylphenylcarbamide, $NHPh\cdot CO\cdot N(C_{10}H_7)\cdot N_2Ph$, forms microscopic needles melting at 123°. When treated with acids, *naphthylphenylcarbamide*, $C_{17}H_{14}N_2O$, is formed. This compound is also prepared by the action of phenyl cyanate on β-naphthylamine; it crystallises in short, white, lustrous prisms melting at 220—221°.

Diazoamidotoluene-β-naphthalene, $C_{17}H_{15}N_3$, is prepared by the action of β-diazonaphthalene chloride (1 mol.) on paratoluidine (1 mol) suspended in a solution of sodium acetate. It crystallises from benzene in slender, bright-yellow needles melting at 131—132°.

Paradiazotoluene-β-naphthylphenylcarbamide,

$$NHPh\cdot CO\cdot N(C_{10}H_7)N_2\cdot C_7H_7,$$

forms slender, white needles melting at 110°.

Parabromodiazo-amidobenzenetoluene, $C_7H_7\cdot N_2\cdot NH\cdot C_6H_4Br$, forms long, yellow, lustrous needles melting at 126°. *Paradiazotolueneparabromodiphenylcarbamide* forms slender needles, and melts at 124°; when decomposed by acid, it yields *parabromodiphenylcarbamide* melting at 245°, and other products.

Diazobenzeneparabromodiphenylcarbamide, $C_{19}H_{15}BrNO_4$, is obtained by the action of phenyl cyanate on parabromodiazoamidobenzene; it melts at 115°, and yields parabromodiphenylcarbamide when decomposed by acids.

Diazobenzeneparabromophenylparatolylcarbamide, from phenyl cyanate and parabromodiazoamidobenzene, melts at 138°.

Parabromodiazoamidobenzene-β-naphthalene, $C_{16}H_{12}BrN_3$, is obtained by the action of β-diazonaphthalene chloride on parabromaniline in presence of sodium acetate, and crystallises from benzene in yellow prisms melting at 164°.

β-Diazonaphthaleneparabromodiphenylcarbamide,

$$NHPh\cdot CO\cdot N(C_6H_4Br)\cdot N_2\cdot C_{10}H_7,$$

forms white needles melting at 139—140°. When boiled with acids, parabromophenol, β-naphthol, and parabromodiphenylcarbamide are formed.

Paranitrodiphenylcarbamide, $NHPh\cdot CO\cdot NH\cdot C_6H_4\cdot NO_2$, obtained by the action of phenyl cyanate on paranitrodiazoamidobenzene, crystallises from hot dilute alcohol in bright-yellow crystals, melting at 202°. It is decomposed by potash into carbonic anhydride, aniline, ammonia, and paranitrophenol.

Diazobenzeneparanitrodiphenylcarbamide,

$$NHPh\cdot CO\cdot N(C_6H_4\cdot NO_2)\cdot N_2Ph,$$

is formed when phenyl cyanate is added to an ethereal solution of paradinitrodiazoamidobenzene. It crystallises in groups of white needles melting at 115°, and dissolves in hot benzene with evolution of gas and formation of paranitrodiphenylcarbamide.

Metanitrodiazoamidobenzene, $C_{12}H_{10}N_4O_2$, crystallises in yellow, pointed, thick prisms melting at 131°.

Diazobenzenemetanitrodiphenylcarbamide, $C_{19}H_{15}N_5O_3$, separates in slender, branched needles melting at 104°, when an ethereal solution of the diazo-compound is warmed with phenyl cyanate and treated with light petroleum.

Metanitrodiazoamidobenzenetoluene, $C_{13}H_{12}N_4O_2$, crystallises in lustrous, gold-coloured needles melting at 107°. When the ethereal solution is warmed with phenyl cyanate, *paradiazotoluenemetanitrodiphenylcarbamide*, $NHPh\cdot CO\cdot N(C_6H_4\cdot NO_2)\cdot N_2\cdot C_7H_7$, is obtained; this crystallises in white needles melting at 96°. When the solution in benzene is boiled, metanitrodiphenylcarbamide melting at 197° is formed.

Paranitroparabromodiazoamidobenzene, $C_6H_4Br\cdot N_2\cdot NH\cdot C_6H_4\cdot NO_2$, crystallises in orange-red, lustrous needles melting at 184°.

Parabromodiazobenzeneparanitrodiphenylcarbamide,

$$NHPh\cdot CO\cdot N(C_6H_4\cdot NO_2)\cdot N_2\cdot C_6H_4Br,$$

forms small crystals melting at 129°. When treated with dilute sulphuric acid, paranitrodiphenylcarbamide (m. p. 202°) is formed.

Metanitroparabromodiazoamidobenzene, $C_6H_4Br\cdot N_2\cdot NH\cdot C_6H_4\cdot NO_2$, crystallises in groups of yellow needles melting at 155°.

Parabromodiazobenzenemetanitrodiphenylcarbamide,

$$NHPh\cdot CO\cdot N(C_6H_4\cdot NO_2)\cdot N_2\cdot C_6H_4Br,$$

forms microscopic needles melting at 134°. Sulphuric acid acts on it, yielding metanitrodiphenylcarbamide melting at 197°.

Metanitrometabromodiazoamidobenzene, $C_6H_4Br\cdot N_2\cdot NH\cdot C_6H_4\cdot NO_2$, prepared from metanitrodiazobenzene chloride and metabromaniline, crystallises in yellow needles melting at 106°.

Metabromodiazobenzenemetanitrodiphenylcarbamide,

$$NHPh\cdot CO\cdot N\cdot(C_6H_4\cdot NO_2)\cdot N_2\cdot C_6H_4Br,$$

melts at 128°. When decomposed by sulphuric acid, metanitrodiphenylcarbamide (m. p. 196°) is formed. N. H. M.

New Phenylhydrazine Salts. By W. H. Richardson (*Chem. News*, **58**, 39—40).—The author has prepared phenylhydrazinehydroxyazonaphthalenesulphonic acid by adding aqueous phenylhydrazine hydrochloride to a solution of hydroxyazonaphthalenesulphonic acid acidified with hydrochloric acid; the brown, flocculent precipitate which first forms becomes scarlet and crystalline on adding the hydrazine. The yield is quantitative; the substance is sparingly soluble in water, easily in alcohol or acetic acid, but is decomposed by boiling with dilute acids or alkalis. The corresponding barium, calcium, and magnesium salts were prepared.

Phenylhydrazine dinitro-α-naphtholsulphonate is obtained as a crystalline precipitate when aqueous phenylhydrazine hydrochloride is added to 1 gram of naphthol-yellow dissolved in a litre of water. It is sparingly soluble in boiling water, and crystallises from that medium in light brown needles. D. A. L.

Action of Nitroso-bases on Phenylhydrazine. By O. Fischer and L. Wacker (*Ber.*, **21**, 2609—2617).—The analyses of the compound obtained by the action of phenylhydrazine on nitrosoaniline point rather to the formula $C_{12}H_{12}N_4O$ than to that previously given (this vol., p. 460); paraphenylenediamine is formed in the reaction.

Nitrosodimethylaniline (3 mols.) reacts with phenylhydrazine acetate (2 mols.) with formation of the compound $C_{14}H_{16}N_4O$. This is obtained in splendid, yellow needles or plates by carefully adding water to the alcoholic solution; it melts at 103° with decomposition, and detonates when heated quickly. When the solution in chloroform is heated, it decomposes with explosive violence and liberation of carbonic anhydride.

Free phenylhydrazine reacts with nitrosodimethylaniline dissolved in ether with formation of parazodimethylaniline, dimethylphenylenediamine, and an oil. When an alcoholic solution is used, a small amount of parazodimethylaniline, together with some dimethylphenylenediamine and *dimethylamidodiphenylamine*, $NHPh{\cdot}C_6H_4{\cdot}NMe_2$, are obtained. The latter crystallises from light petroleum in needles which melt at 130° and sublime below 100°; it is readily soluble in dilute hydrochloric, less in dilute sulphuric acid. When the dilute acid solution is treated with sodium nitrite, the *nitrosamine*, $C_{14}H_{15}N_3O$, is formed. This crystallises in slender, pale yellow needles which melt at 116° with decomposition.

Benzenediazonitrosodiphenylamine, $C_{18}H_{16}N_4O$, obtained by the action of phenylhydrazine on nitrosodiphenylamine in acid solution, crystallises in lustrous, gold-coloured plates, which become greenish when exposed to light; it melts with decomposition at 112° and detonates when quickly heated.

Free phenylhydrazine dissolved in ether acts on nitrosodiphenylamine, yielding amidodiphenylamine melting at 75°, and *azoxydiphenylamine*, $C_{24}H_{20}N_4O$, which crystallises in splendid, gold-coloured plates melting at 173°. When an alcoholic solution is used instead of an ethereal solution, Calm's paradiphenylphenylenediamine (Abstr., 1884, 594) is formed. This melts at 145° (not 152°).

Parazoxyphenol, $ON_2(C_6H_4{\cdot}OH)_2$, is formed, together with paramido-

phenol, when equal parts of nitrosophenol and phenylhydrazine are treated with ether (5—6 parts). It crystallises from dilute alcohol in pale-yellow, lustrous plates with 1 mol. H_2O; the hydrate has probably the constitution $OH{\cdot}C_6H_4{\cdot}N(OH){\cdot}N(OH){\cdot}C_6H_4{\cdot}OH$.

N. H. M.

Action of Secondary Aromatic Amines and Hydrazines on Bromacetophenone. By J. Cullmann (*Ber.*, **21**, 2595—2597).—When bromacetophenone (1 mol.) and methylaniline (2 mols.) are mixed, heat is developed, and on cooling the whole solidifies; the product contains Staedel and Siepermann's phenacylmethylanilide (Abstr., 1881, 722). When this is heated with zinc chloride (5 parts) at 180°, 1′ : 2′-methylphenylindole melting at 101° is formed (Degen, Abstr., 1887, 145).

When bromacetophenone and methylaniline are heated to boiling, a violent reaction takes place with formation of 2′-phenylindole and another compound, probably 1′ 2′-methylphenylindole.

Phenylglyoxalmethylphenylosazone, $NMePh{\cdot}N{:}CPh{\cdot}CH{:}N{\cdot}NMePh$, is formed when a solution of methylphenylhydrazine (3 mols.) dissolved in alcohol (5 parts) at 0° is treated with bromacetophenone (1 mol.). After some hours, the whole becomes dark-red, and is then kept for a week at the ordinary temperature; it is filtered, washed with ether, and crystallised from hot alcohol, from which it separates in yellowish-red prisms melting at 151°.

N. H. M.

Osazones. By H. v. Pechmann (*Ber.*, **21**, 2751—2762).—To detect the presence of an osazone, the substance is moistened with alcohol, gently warmed with a little ferric chloride, and the mixture shaken with ether; if an osazone is present, the ether is coloured red or reddish-brown. This reaction depends on the fact that the osazones of simple 1.2-diketo-derivatives are very readily converted into coloured, crystalline substances when treated with oxidising agents. Only those osazones which are derived from simple, fatty diketo-compounds or those which contain but one aromatic radicle give this reaction. Tartrazine and the osazones of benzil, acetylglyoxylic acid, dehydroxytartaric acid and glucose do not show the reaction. Acetylglyoxylic acid osazone yields pyrazolone-derivatives when treated as described above. The coloured oxidation products of osazones, which the author names *osotetrazones*, are neutral substances containing 2 atoms of hydrogen less than the osazones.

Diacetylosazone is best prepared as follows:—A mixture of 14·4 parts of methyl acetoacetate, 250 parts of water, and 48 parts of soda (1 : 5) is kept for about 24 hours, 7 parts of sodium nitrite added, and finally dilute sulphuric acid gradually poured into the well-cooled solution until the liquid just colours tropæolin paper violet, the whole being well agitated. After adding soda until the solution becomes yellow, it is acidified with acetic acid and mixed with 10·8 parts of phenylhydrazine in 30 parts of 50 per cent. acetic acid, the whole being well stirred. The *hydrazoxime*, melting at 158°, which separates from the solution is collected, washed, dried, and heated at 100° for about an hour with two-thirds of its weight of phenylhydrazine dissolved in 50 per cent. acetic acid. The osazone, which separates

almost completely, is collected, washed, and dried. 144 grams of methyl acetoacetate yield 180 grams of the osazone.

Diacetylosotetrazone, $<^{CMe:N\cdot NPh}_{CMe:N\cdot NPh}>$, is prepared by heating the osazone (5 parts) with potassium dichromate (4 parts), water (20 parts), and 50 per cent. acetic acid (5 parts), until the solution begins to become turbid. It crystallises from boiling alcohol in dark claret-coloured, flat needles, melts at 169° with decomposition, and is soluble in chloroform and benzene, more sparingly soluble in ether, acetone, and alcohol, almost insoluble in glacial acetic acid, and insoluble in water; the solutions are brownish-red. It dissolves in concentrated sulphuric acid with a blue coloration which immediately changes to a dirty brown; when warmed with phenylhydrazine, it yields a red solution, which at a higher temperature becomes colourless and contains the osazone, which is precipitated in a pure state on adding alcohol.

Methylglyoxalosotetrazone, $<^{CH:N\cdot NPh-}_{CMe:N\cdot NPh}>$, is prepared in like manner from methylglyoxalosazone (Abstr., 1887, 1104) which is best obtained by the method described in the case of diacetylosazone. The yield is quantitative. It crystallises from boiling alcohol in needles, somewhat lighter coloured and rather more readily soluble than, but otherwise similar to, those of the diacetyl-derivative. It melts at 106—107°, decomposes at 124°, and behaves towards sulphuric acid and phenylhydrazine similarly to diacetylosotetrazone. In the preparation of methylglyoxalosazone a crystalline hydrazoxime, $CMe\cdot N_2HPh\cdot CH:NOH$, is obtained.

Glyoxalosotetrazone, prepared from glyoxalosazone, crystallises from alcohol in dark-red plates melting at 152° with decomposition.

When osazones or osotetrazones are boiled with dilute hydrochloric acid, they are decomposed into aniline and neutral, non-volatile, very stable, oily compounds which the author names *osotriazones*.

Dimethylphenylosotriazone, $C_2Me_2N_3Ph$, can be obtained by warming diacetylosotetrazone with 6 to 7 parts of water and 1½ parts of concentrated hydrochloric acid until it is wholly converted into a brown oil, and then distilling the product with steam. An addition of 1½ parts of 50 per cent. ferric chloride solution increases the yield. In this reaction, considerable quantities of diacetylhydrazone are formed, and when the decomposition of the osotetrazone is carried out in alcoholic solution, this hydrazone is the principal product. Dimethylphenylosotriazone can also be prepared by gradually adding phosphoric chloride (40—45 grams) to a chloroform (300 grams) solution of the hydrazoxime referred to above. The reaction product is carefully mixed with water and the chloroform solution separated, washed, filtered, and evaporated. The osotriazone obtained by either of these methods is treated with alkaline potassium permanganate until the colour of the latter is permanent, distilled with steam, extracted with ether, and fractionated under diminished pressure. It is a colourless oil, but solidifies on cooling, forming a crystalline substance which melts at 35° and boils at about 255° (at 192°, 60 mm.). It is almost insoluble in water but soluble in all other liquids. It dissolves

in concentrated acids but is reprecipitated by water. The *picrate* is crystalline. It dissolves unchanged in fuming nitric acid, but on adding concentrated sulphuric acid, it forms a *nitro*-derivative which, after recrystallisation from alcohol, is obtained in white needles melting at 227°. It is very stable towards reducing agents and is not acted on by hydriodic acid and phosphorus at 200°. When treated with sodium and alcohol, it yields small quantities of aniline and other bases. It is acted on by oxidising agents only after prolonged boiling, and when thus treated with alkaline potassium permanganate it yields a crystalline acid, probably bibasic. The constitution of this osotriazone is probably $<\begin{matrix}CMe{:}N\\CMe{:}N\end{matrix}>NPh$.

Methylphenylosotriazone, C_2HMeN_3Ph, is prepared by treating methylphenylosotetrazone with hydrochloric acid as described above. It is a colourless oil, boils at 149—150° (60 mm.), and resembles the preceding compound, from which it differs however in the following reactions:—When treated with sodium and alcohol, it forms a deep-red solution which is decolorised on adding water. Salts of diazobenzene produce a brownish-red precipitate in aqueous alcoholic solutions.

Phenylosotriazonecarboxylic acid, $COOH{\cdot}C_2HN_3Ph$, is obtained when methylphenyltriazone is boiled with alkaline potassium permanganate for a long time, and the unaltered oil distilled with steam. It crystallises in needles, melts at 191—192°, and is soluble in alcohol, ether, and chloroform, but almost insoluble in cold water. It distils undecomposed when heated alone, but it is not volatile with steam. When treated with sodium amalgam in the cold, it is readily reduced, yielding aniline and other basic products. The *barium* salt crystallises from water and is sparingly soluble. The *silver* salt, $C_9H_6AgN_3O_2$, is stable, and when distilled, yields an oil which shows the reaction of methylphenyltriazone with alcohol and sodium, and is probably phenylosotriazone. F. S. K.

Paratolenylimido Ethyl Ether. By G. Glock (*Ber.*, **21**, 2650—2659).—*Paratolenylimido ethyl ether hydrochloride*,

$$OEt{\cdot}C(C_7H_7){:}NH,HCl,$$

prepared by saturating an alcoholic ethereal solution of paratolunitrile with hydrogen chloride, melts at 161° with decomposition, is readily soluble in alcohol, insoluble in ether and benzene, and is decomposed by water into ethyl paratoluate and ammonium chloride. When heated at 200°, it yields ethyl chloride and toluamide. The *platinochloride*, $(C_{10}H_{13}NO)_2,H_2PtCl_6 + 2H_2O$, is crystalline but unstable. The free *base* is a liquid; it is decomposed into tolunitrile and ethyl alcohol when distilled, and when kept for a long time, a polymeric compound separates in long needles; this melts at 260°, and is sparingly soluble in hot alcohol and insoluble in water.

Paratolenylimidoacetate, $OAc{\cdot}C(C_7H_7){:}NH$, prepared by heating the hydrochloride of the imidoether with acetic anhydride and sodium acetate, crystallises in prisms, melts at 147°, is readily soluble in alcohol, sparingly soluble in water, and insoluble in ether.

Tolenylamidine hydrochloride, $NH_2{\cdot}C(C_7H_7){:}NH,HCl$, crystallises in prisms with $\frac{1}{2}$ mol. H_2O, melts at 213°, and is readily soluble in water and alcohol but insoluble in ether. The free *base* crystallises in nacreous plates, melts at 101—102°, and is soluble in alcohol and ether. The *platinochloride*, $(C_8H_{10}N_2)_2,H_2PtCl_6$, crystallises in bright yellow needles, melts at 225°, and is sparingly soluble in water. The *nitrate*, $C_8H_{10}N_2,HNO_3 + 2H_2O$, is crystalline, melts at 95°, and is readily soluble in water. The *sulphate* $(C_8H_{10}N_2)_2,H_2SO_4 + 2H_2O$, crystallises in prisms melting at 240°, and is readily soluble in water.

Dimethyltolenylamidine hydrochloride, $NHMe{\cdot}C(C_7H_7){:}NMe,HCl$, the reaction product of tolenylimido ethyl ether hydrochloride and methylamine, crystallises in long, silky needles, melts at 200°, and is readily soluble in water and alcohol. The *platinochloride*,

$$(C_{10}H_{14}N_2)_2,H_2PtCl_6 + 2H_2O,$$

forms quadratic crystals and melts at 95°. The hydrochloride of the isomeric compound, $NMe_2{\cdot}C(C_7H_7){:}NH$, is obtained when dimethylamine is employed in the place of methylamine in the above reaction. It crystallises in prisms.

Ethyltolenylamidine platinochloride, $(C_{10}H_{14}N_2)_2,H_2PtCl_6 + 4H_2O$, crystallises in long needles and melts at 65°. *Diphenyltolenylamidine*, $NHPh{\cdot}C(C_7H_7){:}NPh$, crystallises in white plates, melts at 168°, and is readily soluble in alcohol, ether and benzene, but insoluble in water.

The compound, $NH[C(NH){\cdot}C_7H_7]_2$, is obtained, together with toluic acid, when paratolenylamidine is boiled with sodium acetate and acetic anhydride. It crystallises in needles, melts at 152—153°, and is almost insoluble in all ordinary solvents.

Tolylmethylhydroxypyrimidine, $C_7H_7{\cdot}C\begin{matrix}N\cdot\!-\!CMe\\ N{:}C(OH)\end{matrix}\!>CH$, is formed when tolenylamidine is heated with ethyl acetoacetate. It crystallises in long needles, melts at 216°, is readily soluble in chloroform and moderately soluble in hot alcohol, but insoluble in water. The *platinochloride*, $(C_{12}H_{12}N_2O)_2,H_2PtCl_6 + 2H_2O$, crystallises in bright yellow needles and melts at 241°. The *picrate*, $C_{12}H_{12}N_2O,C_6H_3N_3O_7$, forms yellow needles, melts at 296°, and is readily soluble in alcohol and hot water, but only sparingly in cold water. The *dichromate*, $(C_{12}H_{12}N_2O)_2,H_2Cr_2O_7 + 7H_2O$, crystallises in yellow plates melting at about 170°. F. S. K.

Phenyleneparadiacetimido Ethyl Ether. By G. Glock (*Ber.*, **21**, 2659—2664).—*Phenyleneparadiacetimido ethyl ether hydrochloride*, $C_6H_4[CH_2{\cdot}C(NH){\cdot}OEt]_2,2HCl$, prepared by treating paraxylylene cyanide with hydrogen chloride in alcoholic solution, crystallises in needles, melts above 240°, and is readily soluble in alcohol but insoluble in benzene and ether. It is hygroscopic and decomposes on exposure to the air or when dissolved in water.

Phenyleneparadiacetamidine hydrochloride,

$$C_6H_4[CH_2{\cdot}C(NH){\cdot}NH_2]_2,2HCl,$$

crystallises in prisms, is readily soluble in water and alcohol, and does not melt when heated to 240°. The free *base* crystallises in plates,

melts at 182°, is readily soluble in benzene, alcohol, and ether, and absorbs carbonic anhydride from the air.

Xylyleneparadimethylhydroxypyrimidine,

$$CH\langle\begin{matrix}C(OH):N\\CMe-N\end{matrix}\rangle C\cdot CH_2\cdot C_6H_4\cdot CH_2\cdot C\langle\begin{matrix}N:C(OH)\\N-CMe\end{matrix}\rangle CH,$$

prepared by treating phenylenediacetimido ethyl ether (1 mol.) with ethyl acetoacetate (2 mols.), is crystalline, melts at 250°, and is insoluble in ordinary solvents.

Amidotolunitrile, prepared from metanitrotolunitrile, crystallises in white plates, melts at 93°, and is readily soluble in alcohol, ether, and benzene, but insoluble in light petroleum.

Homophthalonitrile, prepared from the preceding compound by Sandmeyer's method, crystallises in slender, white needles, melts at 117°, and is readily soluble in alcohol and benzene but insoluble in light petroleum.

Metacyanoparatolenylimido ethyl ether hydrochloride,

$$CN\cdot C_7H_6\cdot C(NH)\cdot OEt,HCl,$$

prepared by passing hydrogen chloride into an alcoholic benzene solution of homophthalonitrile, crystallises in prisms and melts at 199°. F. S. K.

Azophenine and Induline. By O. Fischer and E. Hepp (*Ber.*, **21**, 2617—2624; compare this vol., p. 472).—When azophenine (400 grams) is boiled with alcohol (40 litres) and strong sulphuric acid (2 kilos.) for 10 hours, quinonedianilide (195 grams) and induline (20 grams) are formed (compare Abstr., 1887, 1105). This reaction and v. Bandrowski's synthesis of azophenine from diphenylparazophenylene (this vol., p. 1081) confirm the formula

$$C_6H_2(NPh)_2(NHPh)_2,$$

previously ascribed to azophenine. The constitution of induline obtained by the addition of aniline to azophenine is probably—

$$NPh\cdot C_6H(NHPh)_2\langle\begin{matrix}N-\\NPh\end{matrix}\rangle C_6H_4.$$

When nitrosodiphenylamine (10 grams) is heated with aniline hydrochloride (12 grams) and water (1 litre) for two hours in a water-bath, phenylsafranine (identical with Perkin's pseudomauveïne), a soluble induline, and aniline-black are formed.

Rosinduline, $NPh:C_{10}H_5\langle\begin{matrix}N-\\NPh\end{matrix}\rangle C_6H_4$, is prepared by adding nitrosoethyl- or nitrosophenyl-α-naphthylamine (1 part) to a mixture of aniline hydrochloride (1 part) and aniline (3 to 4 parts), heated to 90°, and afterwards heating the whole for some hours at 110—120°. The product is treated first with water, then with dilute hydrochloric acid, the residue extracted with boiling alcohol, and after being diluted with water, filtered and precipitated with ammonia. It forms lustrous, garnet-coloured plates, which melt at 235°, and dissolve in benzene, but are less soluble in alcohol, and almost insoluble in water. The *platino-*

chloride, $(C_{28}H_{19}N_3)_2,H_2PtCl_6$, forms reddish-yellow crystals; the *hydrochloride* crystallises in thick, reddish-brown prisms with lustrous, gold-coloured surfaces, sparingly soluble in water, more readily in alcohol. The crystals from dilute alcohol contain $1\frac{1}{2}$ mol. H_2O. The *acid sulphate*, $C_{28}H_{19}N_3,H_2SO_4$, crystallises from dilute alcohol in bronze-coloured prisms with 1 mol. H_2O. The *nitrate* forms needles or prims of a bronze-like lustre, and melts at 231°. The *picrate* crystallises in lustrous plates. When the base is heated with hydriodic acid and amorphous phosphorus at 150°, aniline hydriodide and a hydriodide, $C_{22}H_{18}N_2,HI$, are formed. If the reduction takes place at 200°, a large amount of aniline hydriodide is obtained together with a neutral substance which seems to be a mixture of di- and tetra-hydronaphthalene. N. H. M.

Benzotribromanilide. By S. Borrelli (*Gazzetta*, **17**, 527—528).—This compound is obtained on gently heating a mixture of symmetrical tribromaniline with benzoic chloride in molecular proportion until hydrogen chloride ceases to be evolved, washing the product with a warm solution of sodium carbonate, and crystallising the residue from alcohol. *Benzotribromanilide*, $C_6H_3Br_3{\cdot}NHBz$, forms stellate groups of needles melting at 198°.

If the anilide is dissolved in cold nitric acid of sp. gr. 1·5 and the solution poured into much water, a yellowish, flocculent precipitate is produced which is a mixture of nitro-derivatives. By treatment with alcohol, the product may be separated into three portions, one insoluble, another very soluble, and a third moderately soluble. The insoluble portion, which is left as colourless, silky needles, melts at 272—273°, and is a dinitro-derivative, $C_{13}H_6Br_3(NO_2)_2NO$. The fraction which is moderately soluble also crystallises in colourless, silky needles melting at 227°. It is likewise a dinitro-derivative isomeric with the insoluble one. Nothing definite could be separated from the very soluble fraction. C. E. G.

Derivatives of Propylamine. By S. Gabriel and J. Weiner (*Ber.*, **21**, 2669—2679).—*Trimethylenediphthalimide*,

$$CH_2(CH_2{\cdot}N{:}C_6H_4O_2)_2,$$

prepared by heating a mixture of trimethylene bromide (1 mol.) and potassium phthalimide (2 mols.) at about 170°, crystallises in slender, white needles, melts at 197—198°, is sparingly soluble in ether, carbon bisulphide, chloroform, and cold alcohol, moderately soluble in hot glacial acetic acid, and almost insoluble in water and light petroleum. It yields phthalic acid and trimethylenediamine hydrochloride when heated at 180—200° with concentrated hydrochloric acid.

Trimethylenediphthalamic acid, $C_3H_6(NH{\cdot}CO{\cdot}C_6H_4{\cdot}COOH)_2$, prepared by warming the imide with potash, is a white, crystalline substance which begins to decompose at 70°, and seems to be decomposed when boiled with water. It yields phthalic acid and trimethylenediamine when heated with hydrochloric acid. The *silver* salt, $C_{19}H_{16}Ag_2N_2O_6$, crystallises in prisms.

γ-Bromopropylphthalimide, $CH_2Br{\cdot}CH_2{\cdot}CH_2{\cdot}N{:}C_6H_4O_2$, is obtained,

together with trimethylenediphthalimide, by heating a mixture of potassium phthalimide (70 grams) and trimethylene bromide (210 grams) at about 70°. The reaction product is distilled with steam, and the residual oil, which solidifies on cooling, is crystallised from boiling alcohol and extracted with ether. Trimethylenediphthalimide (6 grams) remains, and almost pure brompropylphthalimide (63 grams) is obtained when the ethereal solution is evaporated. It crystallises from light petroleum in long needles, melts at 72—73°, is very sparingly soluble or quite insoluble in water, and only sparingly in light petroleum, but readily in warm alcohol and ether.

γ-Hydroxypropylamine, $OH{\cdot}C_3H_6{\cdot}NH_2$, is formed, together with hydrogen bromide and phthalic acid, when the preceding compound is heated at about 200° with sulphuric acid. The reaction product is freed from phthalic acid by filtration, the filtrate treated successively with barium carbonate and silver carbonate, and the solution of the base evaporated with hydrochloric acid. The *hydrochloride* is a syrup. The *platinochloride*, $(C_3H_9ON)_2,H_2PtCl_6$, crystallises in yellow plates and is readily soluble in water. The *aurochloride*, $C_3H_9ON,HAuCl_4$, is crystalline.

γ-Bromopropylamine hydrobromide, $CH_2Br{\cdot}C_2H_4{\cdot}NH_2,HBr$, is obtained, together with phthalic acid and small quantities of bromethylenediamine hydrobromide, when bromopropylphthalimide is heated at 180—200° with concentrated hydrobromic acid. It crystallises from alcohol in slender needles, and from ether in small plates, melts at 162°, and is readily soluble in water and alcohol. The *picrate*, $C_3H_6Br{\cdot}NH_2,C_6H_3N_3O_7 + \frac{1}{2}H_2O$, begins to melt at 115° and is completely melted at 125°; the anhydrous salt melts at 125—127°.

β-Bromopropylamine picrate can be prepared by saturating aqueous allylamine with hydrogen bromide, evaporating the solution, heating the residual salt with concentrated hydrobromic acid at 100°, and precipitating an aqueous solution of the product with sodium picrate. It forms compact, yellow, anhydrous crystals and melts at 154° with previous softening.

When aqueous γ-bromopropylamine hydrobromide (1 mol.) is treated with cold soda (1 mol.), a strongly alkaline solution of amine-like odour is obtained which, after acidifying, gives no precipitate with potassium bismutho-iodide solution. On heating the alkaline solution at 80° in a closed vessel, it loses its alkaline reaction almost completely, and on adding potassium bismutho-iodide to a small portion, a heavy, cinnabar-red, crystalline precipitate is formed. When this solution is saturated with alkali and distilled with steam, a distillate is obtained which contains two volatile bases but is free from allylamine. The distillate is evaporated to a syrup, the bases separated by adding potash, dried first over potash and then over sodium, and fractionated.

The *base*, C_3H_7N, probably trimethylenimine, distils between 65° and 80°. It has a strongly ammoniacal odour, fumes in the air, and is miscible with water. It combines with carbon bisulphide with explosive violence, but does not show either of Hofmann's reactions for primary amines. The *picrate*, $C_3H_7N,C_6H_3N_3O_7$, crystallises in

compact, yellow needles or granules melting at 166—167°. The *aurochloride*, $C_3H_7N,HAuCl_4$, separates in yellow, fern-like crystals, and is sparingly soluble in cold but readily soluble in hot water. The *platinochloride*, $(C_3H_7N)_2,H_2PtCl_6$, crystallises in orange-yellow needles and melts at 200—203° with decomposition.

The *base*, $C_6H_{14}N_2$, distils between 160° and 167°. It has an amine-like odour, a strongly alkaline reaction, and is miscible with water. It combines with carbon bisulphide with evolution of heat, and gives both the reactions for primary amines. The *picrate*,

$$C_6H_{14}N_2,2C_6H_3N_3O_7,$$

separates from hot water, in which it is sparingly soluble, in indented crystals melting at about 210° with decomposition. The *aurochloride*, $C_6H_{14}N_2,2HAuCl_4$, crystallises in yellow, microscopic plates and is rather sparingly soluble. The *platinochloride*, $C_6H_{14}N_2,H_2PtCl_6$, crystallises in orange-red plates and melts at about 215° with decomposition. The authors will continue their investigation of these bases.

F. S. K.

Action of Potassium Phthalimide on Oxyhalogen Compounds. By C. GOEDECKEMEYER (*Ber.*, **21**, 2684—2692).—*Acetonylphthalimide*, $COMe\cdot CH_2\cdot N:C_8H_4O_2$, the reaction-product of potassium phthalimide and chloracetone, crystallises from boiling water in plates and needles melting at 117°. It is rather easily soluble in alcohol, ether, benzene and acetic acid, but almost insoluble in light petroleum. The *oxime*, $NOH:CMe\cdot CH_2N:C_8H_4O_2$, forms small, prismatic crystals, melting at 172°, and is readily soluble in chloroform, alcohol, and glacial acetic acid, but only sparingly in light petroleum, and almost insoluble in boiling water. The *hydrazide*, $N_2HPh:CMe\cdot CH_2\cdot N:C_8H_4O_2$, separates in yellow, prismatic crystals, melts at 150—152° with decomposition; it is very sparingly soluble in hot water, but readily in most of the ordinary solvents.

Phenacylphthalimide, $COPh\cdot CH_2\cdot N:C_8H_4O_2$, prepared by heating potassium phthalimide with bromacetophenone, crystallises from glacial acetic acid in plates, melts at 167°, and is soluble in alcohol, ether, chloroform, and benzene, but almost insoluble in water and light petroleum. The *hydrazide*, $N_2HPh:CPh\cdot CH_2\cdot N:C_8H_4O_2$, crystallises from glacial acetic acid in slender, orange-yellow needles, melts at 155°, and is soluble in the ordinary solvents except water.

Phenacylphthalamic acid, $COPh\cdot CH_2\cdot NH\cdot CO\cdot C_6H_4\cdot COOH$, is obtained when the imide is treated with warm, alcoholic potash. It crystallises from glacial acetic acid in small needles, melts at 160°, and is soluble in almost all the ordinary solvents except water. The *silver* salt, $C_{16}H_{12}NO_4Ag$, and the copper salt are crystalline. The acid is converted into α-amidoacetophenone hydrochloride and phthalic acid when boiled with concentrated hydrochloric acid.

α-Amidoacetophenone picrate, $COPh\cdot CH_2\cdot NH_2,C_6H_3N_3O_7$, crystallises in long, yellow needles, melting at 175° with decomposition.

Ethyl phthalylamidoacetate, the reaction-product of potassium phthalimide and ethyl chloracetate, crystallises from hot alcohol or boiling water in long, white needles, melts at 112—113°, and is not

hydrolysed when boiled with water (compare Reese, this vol., p. 148). It yields amidoacetic acid, phthalic acid, and ethyl chloride when heated at 200° with concentrated hydrochloric acid.

β-Hydroxytrimethylenediphthalimide, $OH{\cdot}CH(CH_2{\cdot}N{\cdot}C_8H_4O_2)_2$, is obtained in small quantities when potassium phthalimide and epichlorhydrin are heated together at 120°, and in larger quantities by heating the imide with dichlorhydrin at 145—150°. It crystallises from glacial acetic acid in white needles, melts at 204—205°, and is readily soluble in chloroform, moderately soluble in alcohol and benzene, and insoluble in water.

Hydroxytrimethylenediphthalamic acid,

$$OH{\cdot}CH(CH_2{\cdot}NH{\cdot}CO{\cdot}C_6H_4{\cdot}COOH)_2,$$

is obtained in slender, white needles, when the imide is boiled with potash and the reaction-product precipitated with hydrochloric acid. It is very hygroscopic and cannot be recrystallised, as it very readily resinifies. It melts at 120°, and at 128° is reconverted into the imide. Hydroxytrimethylenediamine hydrochloride (compare Claus, *Ann.*, **168**, 36), prepared by evaporating the preceding compound with hydrochloric acid, or by heating the imide with hydrochloric acid at 165°, crystallises in small, white, hygroscopic needles, melts at 184·5° (corr.), and is very readily soluble in water, but insoluble in ether and absolute alcohol. The *picrate*, $C_3H_{10}N_2O,2C_6H_3N_3O_7$, crystallises in yellow needles, and melts at 230° with decomposition. The *platinochloride*, $C_3H_{10}N_2O,H_2PtCl_6$, crystallises in long, orange needles, and is soluble in cold water, but insoluble in alcohol and ether. It is decomposed when heated at about 240°. The *aurochloride* is crystalline and sparingly soluble. The *ferrocyanide* is similar. F. S. K.

Amido-derivatives of Metaxylene. By R. Brömme (*Ber.*, **21**, 2700—2706).—*Metaxylylphthalimide*, $C_6H_4Me{\cdot}CH_2{\cdot}N{:}C_8H_4O_2$, is formed when metaxylyl bromide and potassium phthalimide are heated together at 180—200°. It crystallises from hot alcohol in slender, white, hexagonal needles, melts at 117—118°, and is readily soluble in ether, chloroform, and alkalis.

Metaxylylphthalamic acid, $COOH{\cdot}C_6H_4{\cdot}CO{\cdot}NH{\cdot}C_8H_9$, prepared by boiling the imide with soda, crystallises from hot alcohol, ether, or glacial acetic acid in slender needles melting at 131°. The *silver* salt, $C_{16}H_{14}AgNO_3$, is readily soluble in boiling water.

Metaxylylamine hydrochloride, $C_6H_4Me{\cdot}CH_2{\cdot}NH_2,HCl$, is obtained, together with phthalic acid, when the imide is heated at 180—200° with concentrated hydrochloric acid. It forms compact, rhombic crystals or slender needles. The free *base* is a colourless, strongly basic-smelling liquid, boiling at 201—202° (753 mm.); it is miscible with ether and alcohol, and absorbs carbonic anhydride from the air, forming a crystalline compound. The *platinochloride*, $(C_9H_{11}N)_2,H_2PtCl_6$, crystallises from hot water in golden-yellow plates melting at 212°. The *sulphate*, melting at 248°, and the *oxalate*, $(C_9H_{11}N)_2,C_2H_2O_4$, melting at 172°, crystallise in plates, and are readily soluble in water.

The *picrate* crystallises from ether or alcohol in concentrically grouped needles melting at 156°.

Metaxylylthiocarbamide, $C_6H_4Me\cdot CH_2\cdot NH\cdot CS\cdot HN_2$, the reaction-product of the hydrochloride of the amine and potassium thiocyanate, crystallises in white needles melting at 112°. *Dimetaxylylthiocarbamide*, $CS(C_6H_4Me\cdot CH_2\cdot NH)_2$, prepared by boiling an alcoholic solution of the amine with carbon bisulphide, crystallises in needles, melts at 97°, and is insoluble in water, acids, and alkalis. It has a slight smell of radishes.

Metaxylylthiocarbimide, $C_6H_4Me\cdot CH_2\cdot N{:}CS$, is formed when a solution of the amine is evaporated with carbon bisulphide, the reaction-product mixed with mercuric chloride and distilled, or when dixylylcarbamide is heated with syrupy phosphoric acid. It yields xylylthiocarbamide when warmed with ammonia.

Metaxylylcarbamide, $C_6H_4Me\cdot CH_2\cdot NH\cdot CO\cdot NH_2$, crystallises from alcohol in long needles, and melts at 148°.

Dimetaxylylcarbamide, $CO(NH\cdot CH_2\cdot C_6H_4Me)_2$, prepared by treating the amine with carbonyl chloride in ethereal solution, crystallises from alcohol or ether in slender needles melting at 137°.

Metaxylylphenylcarbamide, $NHPh\cdot CO\cdot NH\cdot CH_2\cdot C_6H_4Me$, the reaction-product of the amine and carbamide, crystallises from alcohol, ether, or chloroform in moss-like needles, melts at 131°, and is readily soluble in benzene and glacial acetic acid.

Benzoylmetaxylylamine, $COBz\cdot NH\cdot CH_2\cdot C_6H_4Me$, prepared by heating the amine with benzoic chloride, crystallises from alcohol in white plates, melts at 69°, and is readily soluble in chloroform, acetone, glacial acetic acid, and benzene. The *acetyl*-derivative is an oily liquid boiling at 235—240°.

Metaxylylenediphthalimide, $C_6H_4(CH_2\cdot N{:}C_6H_4O_2)_2$, prepared by heating metaxylylene bromide (26 grams) with potassium phthalimide (45 grams) at 180—200°, crystallises from glacial acetic acid in small needles melting at 237°.

Metaxylylenediamine hydrochloride, $C_6H_4(CH_2\cdot NH_2)_2,2HCl$, is obtained, together with phthalic acid, when the imide is heated at 200—220° with concentrated hydrochloric acid. It crystallises from hot water in long needles, and is readily soluble in dilute alcohol. The *platinochloride*, $C_6H_4(CH_2\cdot NH_2)_2,H_2PtCl_6$, crystallises in compact, yellow plates. The free *base* is a colourless, basic-smelling liquid, boiling at 245—248° (756·5 mm.); it is miscible with alcohol and ether, but only partially miscible with water. When boiled with access of air, it absorbs carbonic anhydride, forming a white, crystalline compound. The *picrate*, $C_6H_4(CH_2\cdot NH_2)_2,2C_6H_3N_3O_7$, crystallises in yellow, scaly plates, and decomposes at 185—190° without melting. The *diacetyl*-derivative, $C_6H_4(CH_2\cdot NHAc)_2$, is a white, crystalline compound, melting at 118—119°, and readily soluble in benzene, ether, alcohol, chloroform, and hot water. F. S. K.

Benzoic Chloride as a Reagent. By L. v. Udránszky and E. Baumann (*Ber.*, **21**, 2744—2751).—When dilute aqueous solutions of fatty diamines are shaken with benzoic chloride and soda, the diamine is almost completely converted into an insoluble dibenzoyl-derivative,

which is easily separated from benzamide and other nitrogenous products by dissolving the precipitate in alcohol and pouring the solution into a large volume of water. After keeping for a short time, the dibenzoyl-derivative crystallises from the solution (compare Baumann, Abstr., 1887, 228).

A solution of 1 gram of ethylenediamine in 500 c.c. of water gave 3·5 grams of crystalline dibenzoyl-derivative when shaken with 10 grams of benzoic chloride and 80 c.c. of soda (about 10 per cent.), and further quantities can be obtained from the filtrate by repeating the operation. The mono-benzoyl-derivative is not formed in this reaction. A distinct precipitate is obtained when a solution of 5 mgrams. of ethylenediamine hydrate in 100 c.c. of water is mixed with a few drops of the reagents, and a solution containing 0·053 gram in 100 c.c. of water gave 0·133 gram of the pure dibenzoyl-derivative.

Pentamethylenediamine and tetramethylenediamine are precipitated from dilute solutions even more completely than ethylenediamine, and the dibenzoyl-derivatives can be obtained quite pure by recrystallising once or twice from alcohol or ether.

Dibenzoylpentamethylenediamine, $C_5H_{10}(NHBz)_2$, crystallises in long needles and plates which melt at 130°; it is readily soluble in alcohol, and moderately in ether, but insoluble in water. It is not acted on by hot dilute acids or alkalis, and when dissolved in concentrated hydrochloric acid it is only changed after prolonged boiling. A solution of 0·00788 gram of pentamethylenediamine in 100 c.c. of water gave 0·0218 gram of the dibenzoyl-derivative when shaken with benzoic chloride (5 c.c.) and 40 c.c. of soda (10 per cent.), and kept for 24 hours; in a second experiment with a similar solution only 0·0142 gram of dibenzoyl-derivative was obtained.

By means of this reagent, the authors succeeded in isolating from the urine of a patient suffering from cystinuria and inflammation of the bladder, pentamethylenediamine, tetramethylenediamine, and a diamine which differed from pentamethylenediamine only in this, that its platinochloride is more readily soluble. A mixture of diamines, consisting chiefly of tetramethylenediamine, was also obtained from the fæces of the patient. Cystine was also invariably found in the urine, and it is probable that the quantity of diamines formed stands in some relation to the amount of cystine produced.

Normal urine and normal fæces do not contain even traces of diamines.

Cystine, dissolved in urine, can be isolated by means of benzoic chloride (compare Goldmann and Baumann, *Zeit. Physiol. Chem.*, **12**, 254).

In the decomposition of albumin by acids, alkalis, and soluble ferments, neither cystine nor diamines are formed, but when the decomposition is caused by bacteria, diamines are soon produced, and can be shown to be present by the benzoic chloride reaction.

Compounds which contain two amido-groups combined with one and the same carbon-atom, do not yield benzoyl-derivatives when shaken with benzoic chloride and soda. F. S. K.

Derivatives of Ethyl Benzoylacetate. By R. STIERLIN (*Ber.*, **21**, 2798).—The compounds, $C_{14}H_{12}N_2O$ (m. p. 129°) and $C_{15}H_{12}N_2O_3$ (m. p. 142°), described by the author (this vol., p. 1089), have been previously obtained by Bamberger (Abstr., 1886, 62).

Cyanacetates. By A. HALLER (*Compt. rend.*, **107**, 104—107).—Ethyl cyansodacetate suspended in absolute alcohol and an acid chloride dissolved in anhydrous ether are mixed together in equal molecular proportions, and the products separated in the manner already described (Abstr., 1887, 1029).

Ethyl orthotoluylcyanacetate, $C_6H_4Me{\cdot}CO{\cdot}CH(CN){\cdot}COOEt$, forms modified rectangular prisms, which melt at 35·2° and remain in superfusion for a long time. It is insoluble in water, but dissolves in ethyl alcohol and ether, and the solutions give with ferric salts a red coloration. When heated with excess of water, it splits up into carbonic anhydride, ethyl alcohol, and orthomethylcyanacetophenone. The calcium-derivative of the ethereal salt crystallises with 4 mols. H_2O in small white needles, slightly soluble in water, but very soluble in ethyl alcohol.

Ethyl phenylacetocyanacetate, $CH_2Ph{\cdot}CO{\cdot}CH(CN){\cdot}COOEt$, isomeric with the preceding compound, is a yellowish oil which cannot be distilled without decomposition, and becomes viscous but does not solidify at −60°. Its solutions give a very intense red coloration with ferric salts. When heated with water, it yields carbonic anhydride, alcohol, and fusible crystals soluble in water and in ether. Its barium-derivative crystallises in white nodules, slightly soluble in water, and very soluble in alcohol. The silver-derivative is white, and insoluble in water.

Ethyl cinnamylcyanacetate crystallises in yellowish needles, which melt at 104°. It is insoluble in water, but slightly soluble in alcohol. The solution has a feebly acid reaction, but decomposes carbonates; with ferric salts it gives a red coloration. When heated with water, the ethereal salt decomposes into alcohol, carbonic anhydride, and a reddish, viscous mass which yields no crystallisable substance.

Ethyl dicinnamylcyanacetate, $CN{\cdot}C(CO{\cdot}CH{:}CHPh)_2{\cdot}COOEt$, is obtained as a secondary product in the preparation of the preceding compound, from which it is separated by its insolubility in alkaline carbonates and in ether. It crystallises in yellowish, silky needles.

C. H. B.

Decomposition of Quinic Acid by Dilute Hydrochloric Acid. By K. CHODOUNSKI (*Chem. Centr.*, 1888, 1029, from *Listy Chem.*, **12**, 267—269).—Quinic acid was heated in sealed tubes with 3 per cent. hydrochloric acid at 100—120°. On opening the tubes, carbonic anhydride was evolved under considerable pressure, and in the solution phenol, quinol, and parahydroxybenzoic acid could be detected, but not trimethylbenzene.

J. W. L.

Phenaceturic Acid and its Derivatives. By E. HOTTER (*J. pr. Chem.* [2], **38**, 97—117).—To synthesise phenaceturic acid by the action of phenylacetic anhydride on glycocine, the former is dissolved in benzene and digested with very finely powdered glycocine for

10 hours in a reflux apparatus. After evaporating the benzene, the mass is extracted with ether, dissolved in hot water, decolorised, and allowed to evaporate; it is recrystallised from alcohol. The method previously published by the author (Abstr., 1887, 368) gives a better yield if the reaction is allowed to take place at −15°.

Synthetical phenaceturic acid, which is identical with the natural product, crystallises from hot water in white laminæ, and from alcohol and acetic ether in cubical crystals, melting at 143°, and decomposing completely at 190—200°. It is very insoluble in pure ether, sparingly so in cold water, in hot benzene, and in chloroform; very soluble in hot water, alcohol, and ethyl acetate.

If the aqueous mother-liquor from the phenaceturic acid crystals is evaporated, a mixture of this acid with a new acid is left, which can be separated by recrystallisation from alcohol. The new acid, $COOH\cdot CH_2\cdot NH\cdot CO\cdot CH_2\cdot NH\cdot CO\cdot CH_2Ph$, analogous to Curtius' hippurylamidoacetic acid (Abstr., 1883, 339), melts at 173—174°, and crystallises in very thin laminæ, insoluble in ether, freely soluble in hot water, alcohol, and aqueous alkali.

The calcium, silver, copper, lead, and zinc salts, are described.

Methyl phenaceturate, $C_{10}H_{10}NO_3Me$, is obtained by the action of methyl iodide on the silver salt; it crystallises in long, silky needles, melting at 86·5°, easily soluble in hot alcohol, chloroform, warm ether, and benzene; it is insoluble in carbon bisulphide. The ethyl salt has been previously described (Abstr., 1887, 369).

Normal propyl phenaceturate crystallises in large rectangular plates, melting at 31°.

Phenaceturamide, $CH_2Ph\cdot CO\cdot NH\cdot CH_2\cdot CONH_2$, is obtained when methyl phenaceturate (1 part) is allowed to remain in contact with a 36 per cent. solution of ammonia (4 to 5 parts). It crystallises in six-sided pearly tables, melting at 174°, insoluble in cold ether and benzene, easily soluble in hot water and alcohol. By digesting an aqueous solution with yellow mercuric oxide, the *mercury* compound is formed, and falls as a crystalline precipitate on cooling.

Nitrophenaceturic Acid, $NO_2\cdot C_6H_4\cdot CH_2\cdot CO\cdot NH\cdot CH_2\cdot COOH$.—Phenaceturic acid (1 part) is allowed to remain in a mixture of nitric acid of sp. gr. 1·33 (4 parts) and concentrated sulphuric acid (6 parts), cooled to 0°; the excess of the acids is filtered off, the nitro-compound dissolved in ammonia, and the solution evaporated. After decomposing the residue of ammonium salt with acid, the nitrophenaceturic acid thus obtained is recrystallised from hot water. It forms very slender needles, melting at 173°, insoluble in boiling benzene and ether, sparingly soluble in cold water and alcohol. The silver and zinc salts are described. The acid gives paranitrophenylacetic acid by prolonged digestion with strong hydrochloric acid.

Amidophenaceturic acid is obtained by dissolving the nitro-acid in yellow ammonium sulphide, passing hydrogen sulphide through the solution, heating on a water-bath until all the ammonium sulphide is decomposed, filtering, and precipitating the filtrate with dilute hydrochloric acid. The amido-acid, recrystallised from alcohol, forms rhomboidal laminæ, soluble in hot water and alcohol.

The author failed to obtain phenacetylglycollic acid by the action of

chlorine on an alkaline solution of phenaceturic acid, and of nitrous acid on an acid solution of phenaceturic acid. Nor could it be synthesised from calcium glycollate and phenylacetic chloride.

The author took phenaceturic acid for some days, but hippuric acid only was found in the urine. A. G. B.

Bromophthalic Acid. By I. Guareschi (*Gazzetta*, **18**, 10—13).—This acid, the *α*-monobromophthalic acid, $C_6H_3Br(COOH)_2$, is obtained from dibromonaphthalene [Br : Br = 1 : 4'] by oxidation; for this purpose, the dibromonaphthalene (2 grams) is dissolved in glacial acetic acid (30 c.c.) a solution of chromic anhydride (4 grams) in acetic acid (20 c.c.) is added, and the mixture heated in a water-bath for a quarter of an hour. It is then diluted with 10 times its volume of water, filtered from the slight yellow precipitate which is formed, and evaporated at 100°. The residue is again evaporated with water, heated with sodium carbonate solution, filtered, and the filtrate acidified with sulphuric acid and repeatedly extracted with ether. On evaporating the ether, the acid is left as a white crystalline residue, which is purified by repeated crystallisation from water. It forms colourless, rhombic prisms, which melt at 178·5° with evolution of water-vapour, and leave the anhydride, which melts at 133—134°. This acid is soluble in water. The *barium salt*, $C_6H_3Br(COO)_2Ba$, obtained from the ammonium salt by double decomposition, crystallises in iridescent laminæ, which are only sparingly soluble in water. The *lead salt* and *silver salts* are white precipitates.

Monobromophthalic anhydride, $C_6H_3Br<\begin{matrix}CO\\CO\end{matrix}>O$, sublimes in lustrous needles, and is identical with that previously obtained by the author from bromonitronaphthalene (m. p. 122·5°), by treatment with potassium permanganate, and by Meldola from dibromonaphthylamine (Trans., 1885, 513). Treated with sulphuric acid and phenol, the anhydride furnishes a brominated phthaleïn, which gives a fine violet-purple coloration with potash.

As Nourrison (*Ber.*, **20**, 1016) has described the *β*-acid, the melting point of which is 168°, Faust and v. Pechmann's acid, melting at 138—140°, is probably a mixture of the *α*- and *β*-acids. C. E. G.

***α*-Benzylhomo-orthophthalic Acid.** By G. Eichelbaum (*Ber.*, **21**, 2679—2683).—*α-Benzylhomo-orthophthalonitrile*,

$$CN\cdot C_6H_4\cdot CH(C_7H_7)\cdot CN,$$

is prepared by treating an alcoholic solution of cyanobenzyl cyanide (1 mol.) and benzyl chloride (1 mol.) with potash (1 mol.). It crystallises from alcohol in colourless plates, melts at 109—110°, and distils above 300°. It is readily soluble in benzene, chloroform, acetone, and hot alcohol, but almost insoluble in cold alcohol, and insoluble in water, alkalis, and acids.

Benzylhomo-orthophthalamide, $CONH_2\cdot C_6H_4\cdot CH(C_7H_7)\cdot CONH_2$, prepared by dissolving the nitrile in concentrated sulphuric acid, crystallises from boiling alcohol in small plates, melts at 224° with evolution

of ammonia, and is soluble in alcohol, benzene, and chloroform, but insoluble in water and alkalis.

Benzylhomo-orthophthalimide, $C_6H_4<\begin{matrix}CH(C_7H_7)\\CO\cdot NH\cdot CO\end{matrix}>$, separates in needles when the amide is heated at 100° with concentrated hydrochloric acid. It melts at 176°, boils above 300°, and is readily soluble in acetic acid, alcohol, and benzene, but insoluble in ether, chloroform, acetone, and light petroleum. It is insoluble in acids, but dissolves in alkalis with a yellow coloration, and on adding more alkali, a yellow, crystalline substance is precipitated.

Benzylhomo-orthophthalic acid, $COOH\cdot C_6H_4\cdot CH(C_7H_7)\cdot COOH$, prepared by heating the corresponding nitrile with strong hydrochloric acid at 200—220°, crystallises in small prisms, melts at 154°, boils above 300°, and is soluble in alcohol and benzene, but insoluble in ether and chloroform. It dissolves in potash with a yellowish coloration.

Benzylchloroxyisoquinoline is obtained, together with an isomeric compound, when benzylhomo-orthophthalimide is heated at 200° with phosphorus oxychloride, and the product extracted with potash. The compound, which is insoluble in potash, crystallises from glacial acetic acid in colourless needles, melts at 234°, and sublimes undecomposed; its constitution is probably $C_6H_4<\begin{matrix}CH(C_7H_7)\\CCl:N\end{matrix}>CO$. The compound, soluble in potash, melts at 195°, and has probably the constitution $C_6H_4<\begin{matrix}C(C_7H_7)\\CO\cdot NH\end{matrix}>CCl$.

F. S. K.

Action of Strong Sulphuric Acid on Diphenylsuccinic Acid; Diphenesuccindone. By W. Roser (*Annalen*, **247**, 152—159).—Reimer (Abstr., 1882, 200) obtained a compound he named dibenzyldicarbonide by the action of strong sulphuric acid on β-diphenylsuccinic acid. The author proposes to call this substance *diphenesuccindone*. It melts at 202°, and is partly converted into isodiphenesuccindone by treatment with sodium hydroxide. The *dioxime* crystallises in needles, and melts with decomposition about 254°.

The *diphenylhydrazide* is sparingly soluble in alcohol and acetic acid; it melts with decomposition between 260° and 270°. Diphenesuccindene, $C_{16}H_{14}$, is obtained by the reduction of diphenesuccindone with phosphorus and hydriodic acid. The hydrocarbon is freely soluble in alcohol and ether, and volatilises in a current of steam. It melts at 100°.

W. C. W.

Paranitrometamidobenzenesulphonic Acid. By E. Eger (*Ber.*, **21**, 2579—2582).—*Paranitrometamidobenzenesulphonic acid*, $NO_2\cdot C_6H_3(NH_2)\cdot SO_3H$, is obtained by heating pure barium metamidobenzenesulphonate, dried at 120—130°, with acetic anhydride (2 parts) for two hours on a water-bath, dissolving in water, and evaporating almost to dryness; alcohol is added to get rid of all the free acetic acid. Barium acetylamidobenzenesulphonate crystallises from the concentrated solution in silky needles, which are washed with alcohol, and dried first at 150° and afterwards at 190°. The barium salt is

dissolved in strong sulphuric acid (5 parts), treated with the calculated amount of nitric acid previously mixed with sulphuric acid (4 parts); the product is poured into ice. The sulphonic acid crystallises in pale yellow needles, rather easily soluble in hot water, sparingly in alcohol, and almost insoluble in ether. The *potassium salt* crystallises readily from water in lustrous, gold-coloured plates; the *sodium salt* forms pale yellow, lustrous needles. When the acid is reduced with zinc-dust and a little hydrochloric acid, a phenylenediamidosulphonic acid is formed, which does not yield an azine-compound with phenanthraquinone, and which gives no yellow coloration with potassium nitrite. Both amido-groups are in the para-position; this is confirmed by the reaction of the diamido-acid with metatoluylenediamine and ferric chloride, which gives rise to a splendid violet coloration. N. H. M.

Amidosulphonic Acids. By G. Pellizzari and V. Matteucci (*Chem. Centr.*, 1888, 1000—1002, from *L'Orosi*, **11**, 145—157).—*Sodium phthalimidophenylsulphonate*, $C_6H_4{:}C_2O_2{:}N{\cdot}C_6H_4{\cdot}SO_3Na$, is obtained by heating equivalent quantities of sodium sulphanilate (amidophenylsulphonate), and anhydrous phthalic acid at 250°. The mass is treated with boiling water, from which the new salt crystallises on cooling in very slender, white needles. It is readily soluble in hot water, much less so in cold. By treatment with warm aqueous alcoholic ammonia, it is decomposed into phthalimide and sodium sulphanilate. The *ammonium* and *barium* salts are prepared in a manner similar to the sodium salt, and are like it in their properties.

Sodium succinimidophenylsulphonate, $C_2H_4{:}C_2O_2{:}N{\cdot}C_6H_4{\cdot}SO_3Na$, is prepared from anhydrous succinic acid and sodium sulphanilate by heating the mixture at 170°, and is purified by recrystallisation from hot water. It forms small, white crystals which are readily soluble in water, and somewhat less so in hot alcohol. The *barium* salt is similar in appearance, but is less soluble.

Carbamide and thiocarbamide react with sodium sulphanilate when heated with it, with formation of sodium carboamidophenylsulphonate and thiocarbamidophenylsulphonate respectively. These may be more readily prepared by heating potassium cyanate and thiocyanate with sulphanilic acid. They are white salts soluble in water.

Potassium phthalimidonaphthylsulphonate, $C_6H_4{:}C_2O_2{:}N{\cdot}C_{10}H_6{\cdot}SO_3K + 3H_2O$, is prepared from potassium α-naphthylaminesulphonate and anhydrous phthalic acid by heating them together to 150°. It consists of small, shining needles.

Potassium succinimidonaphthylsulphonate, $C_2H_4{:}C_2O_2{:}N{\cdot}C_{10}H_6{\cdot}SO_3K$, is prepared in like manner. Both this salt, as also the barium and lead salts, are white and soluble in water.

α-Naphthylsuccinimide, $C_2H_4{:}C_2O_2{:}N{\cdot}C_{10}H_7$, is prepared by heating potassium α-naphthylaminesulphonate with succinic acid at 200°, and extracting the fused mass with water. It forms white, shining crystals which may be distilled without decomposition. By dissolving this in hot aqueous potash, *α-naphthylsuccinamic acid*,

$$COOH{\cdot}C_2H_4{\cdot}NH{\cdot}C_{10}H_7,$$

is formed, and may be precipitated from the alkaline solution by the addition of an acid. Crystallised from alcohol, it forms flattened needles melting at 171°, and is soluble in alcohol, benzene, and acetic acid. The salts of barium, silver, and copper are but little soluble.

β-Naphthylsuccinimide and the corresponding acid are prepared exactly in a similar manner to the last-named compounds, and show similar properties and solubility.

Potassium phthalimidoisethionate, $C_6H_4{:}C_2O_2{:}N{\cdot}C_2H_4{\cdot}SO_3K + \frac{1}{2}H_2O$, is prepared by evaporating equivalent quantities of taurine and potash to dryness, adding anhydrous phthalic acid, and heating at 160°. By extracting the mass with hot water, the new compound is obtained in beautiful, colourless, monoclinic crystals. By melting the potassium salt of taurine with succinic acid, potassium succinimidoisethionate is obtained. J. W. L.

Indene-derivatives. By W. Roser (*Annalen*, **247**, 129—138).—Indene-derivatives are obtained by the action of sulphuric acid on diphenylsuccinic, bromocinnamic, and dichloro- and dibromo-succinic acids (Abstr., 1887, 729), also from ethyl benzylacetoacetate (Abstr., 1887, 836). W. C. W.

Methylindenecarboxylic Acid. By W. Roser (*Annalen*, **247**, 157—166).—The preparation and properties of *γ-methylindene-β-carboxylic acid*, $C_6H_4{<}\begin{matrix}CMe\\CH_2\end{matrix}{>}C{\cdot}COOH$, have already been described by the author (Abstr., 1887, 836). The compound $C_{11}H_{10}O_2,C_2H_4O_2$ is deposited in prisms from a solution of methylindenecarboxylic acid in glacial acetic acid. On exposure to the air, the crystals effloresce, losing acetic acid. The *methyl* salt melts at 78°, and dissolves easily in alcohol and ether. *γ-Methylindene*, $C_{10}H_{10}$, is obtained by heating the acid with soda-lime. It is a highly refractive liquid, and boils at 205—206°. The saturated hydrocarbon methylhydrindene has not been isolated. *γ-Methylindenecarboxylic acid dibromide* melts at 215° with decomposition. The *methyl* salt, $C_{10}H_9Br_2{\cdot}COOMe$, crystallises in white needles, and melts at 157°. The preparation of *α-bromo-α-methylindenecarboxylic acid* has been previously described (*loc. cit.*). The *methyl* salt, $C_{11}H_9Br{\cdot}COOMe$, melts at 98—100°, and is soluble in alcohol and ether. It is formed when bromine is added to a solution of methyl methylindenecarboxylate in chloroform. On removing the solvent by evaporation, hydrogen bromide escapes, and methyl bromomethylidenecarboxylate remains. When a solution of bromomethylidenecarboxylic acid is saturated with hydrogen chloride, the methylic salt of chloromethylidenecarboxylic acid is produced; this is soluble in alcohol and ether, and melts at 84°; sodium hydroxide produces an intense blue coloration in the alcoholic solution. The reduction of methylindenecarboxylic to methylhydrindenecarboxylic acid, $C_6H_4{<}\begin{matrix}CHMe\\CH_2\end{matrix}{>}CH{\cdot}COOH$, has been already described (*loc. cit.*). The new acid melts at 80°, and boils at 300—310°. The silver salt, $C_{11}H_{11}O_2Ag$, and the barium salt, $(C_{11}H_{11}O_2)_2Ba + 2H_2O$, are crystalline. W. C. W.

Dibromindone-derivatives. By W. Roser and E. Haselhoff (*Annalen*, **247**, 138—151).—The authors have previously described (Abstr., 1887, 830) the formation of two isomeric dibromocinnamic acids by the action of bromine on phenylpropiolic acid. The α-acid melts at 139°. It is partially converted into its isomeride, melting at 100°, by recrystallisation from chloroform in presence of free bromine. The β-acid is converted into dibromindone by the action of strong sulphuric acid.

Dibromindone, $C_6H_4<\begin{matrix}CO\\CBr\end{matrix}>Br$, forms orange-coloured needles, and melts at 123°. It dissolves in ether, chloroform, alcohol, and acetic acid, melts under water, and is volatile in a current of steam. When a mixture of α- and β-dibromocinnamic acids is treated with sulphuric acid, the α-acid remains unaltered. *Dibromindonoxime*, $C_6H_4<\begin{matrix}C(NOH)\\CBr{\cdot}CBr\end{matrix}>$, is prepared by boiling an alcoholic solution of dibromindone with hydroxylamine hydrochloride and precipitating the product by the addition of water. It crystallises in silky needles, melts about 198° with decomposition, and forms a crystalline sodium compound. Dibromindone unites with bromine, forming *tetrabromhydrindone*, $C_6H_4<\begin{matrix}CO-\\CBr_2\end{matrix}>CBr_2$. This compound crystallises in prisms, and melts with decomposition at 124°. The alcoholic solution decomposes on boiling, yielding dibromindone. Hydroxylamine hydrochloride acts on the alcoholic solution, forming *tetrabromhydrindonoxime*, $C_9H_5Br_4NO$, melting at 214°.

Dibromindonoxime unites with bromine to form a tribromo-derivative, crystallising in golden needles and melting at 217—218° with decomposition.

Tribromovinylbenzoic acid, $CBr_2{:}CBr{\cdot}C_6H_4{\cdot}COOH$, is formed when an alcoholic solution of tetrabromhydrindone is poured into excess of soda solution. The acid is soluble in alcohol and ether and crystallises in plates. It melts at 196—198°. Dibromodichlorhydrindone, or phenylenedibromodichlorethylene ketone, has been described by Zincke and Frölich (Abstr., 1887, 955). It is reduced to dichlorindone by boiling with alcohol, or by treatment with sulphurous acid.

Bromiodindone is deposited in prisms on boiling an alcoholic solution of dibromindone and potassium iodide. It melts at 163°. Chlorobromindone prepared from dichlorindone melts at 105°. One atom of bromine in dibromindone is easily displaced by the action of amines, yielding anilidobromindone, piperidobromindone, &c.

Bromhydroxyindone is prepared by adding an excess of sodium hydroxide to dibromindone in presence of alcohol. The compound melts at 119°, and is freely soluble in alcohol, benzene, and acetic acid.

Dibromodiketohydrindene and chlorobromodiketohydrindene are formed by acting on an acetic acid solution of bromhydroxyindone with chlorine or bromine. The properties of these derivatives have been described by Zincke and Gerland (this vol., p. 291), and by W. Wislicenus (this vol., p. 1194) respectively. W. C. W.

Application of the Aluminium Chloride Method to the Naphthalene Series. By L. Roux (*Ann. Chim. Phys.*, **12**, 289—358).—The author attempted to prepare methyl-derivatives of naphthalene by Friedel and Craft's method, but without success. In one experiment in which methyl iodide was employed, a very small quantity of a hydrocarbon boiling at about 232° was isolated; it formed a crystalline picrate melting at about 120°.

When aluminium chloride (30—35 grams) is gradually added to a warm mixture of naphthalene (300 grams) and ethylene bromide (150 grams), the whole heated for about two hours, and the product fractionated, the following compounds are obtained:—(1.) A small quantity of a liquid boiling between 300° and 400°, probably consisting of a mixture of hydrides of dinaphthyl. (2.) $\beta\beta$-Dinaphthyl. (3.) A small quantity of a hydrocarbon boiling above 450°, which is probably *dinaphthylnaphthalene*. It crystallises from alcohol in greenish, hexagonal lamellæ, melts at about 300°, is soluble in light petroleum and toluene, and sublimes in small, yellowish scales. (4.) A mixture of α- and β-methylnaphthylene (compare Fittig and Remsen, *Ann.*, **155**, 112; and Schulze, Abstr., 1884, 1183 and 1184).

Ethylnaphthalene is obtained in larger quantities when ethyl bromide is employed instead of the chloride in the method described by Marchetti (Abstr., 1881, 1041; and *Gazzetta*, **11**, 439). When the product of the reaction is fractionated, almost pure ethylnaphthalene can be isolated (compare Fittig and Remsen, *loc. cit.*, and Brunel, Abstr., 1884, 1035).

β-Propylnaphthalene, $C_{10}H_7Pr$, is obtained in an almost pure state when a hot mixture of naphthalene and normal propyl bromide is treated with aluminium chloride and the product fractionated. It is a colourless, highly refractive, aromatic-smelling liquid, boils at 265°, and is readily soluble in benzene, carbon bisulphide, &c., moderately so in alcohol, but insoluble in water. It yields β-naphthoic acid when boiled with dilute nitric acid. The *picrate* crystallises from alcohol or benzene in lemon-yellow needles, and melts at 89—90°.

β-Amylnaphthalene, $C_{10}H_7{\cdot}C_5H_{11}$, is obtained almost pure when amyl chloride (from isobutyl carbinol) is treated in like manner (compare Paternò, *Bull. Soc. Chim.*, **11**, 322, and Leone, Abstr., 1882, 1210). It is a colourless, aromatic-smelling liquid boiling at 288—292°. It yields β-naphthoic acid when oxidised with dilute nitric acid. The *picrate* crystallises from alcohol in lemon-yellow, slender needles melting at 110°.

When amyl chloride or iodide is dropped into a mixture of naphthalene and aluminium chloride at 120°, ethyldimethylmethane and dinaphthyl are formed, but amylnaphthalene is not produced.

When aluminium chloride (5—6 grams) is added to a mixture of benzyl chloride (80 grams) and naphthalene (160 grams) at 100—120°, and the product fractionated, dinaphthyl and a mixture of α- and β-benzylnaphthalenes are obtained. The benzylnaphthalenes can be separated by fractional crystallisation from alcohol (compare Frote, this Journal, 1873, 891; and Miquel, *ibid.*, 1876, ii, 407). When the

mixture of naphthalene and benzyl chloride is heated at 89—90°, only 4 to 5 grams aluminium chloride added, and the reaction stopped in 8 to 10 minutes by pouring the product into water, the α-derivative is obtained mixed with only a small quantity of the β-compound; if the mixture is heated at 160° for an hour, and the aluminium chloride added gradually during this time, the β-derivative is obtained almost free from the α-compound.

α-Benzylnaphthalene crystallises from alcohol in colourless laminæ or prisms, melts at 59°, boils at 350°, and is very readily soluble in benzene, chloroform, and boiling alcohol, but sparingly in cold alcohol. The picrate crystallises from alcohol in golden-yellow needles and melts at 100—101°.

β-Benzylnaphthalene, $C_{10}H_7 \cdot C_7H_7$, separates from alcohol in clinorhombic crystals, melts at 35·5°, and boils at 350°. It is very readily soluble in benzene and boiling alcohol, sparingly in cold alcohol. The *picrate* crystallises in golden-yellow needles melting at 93°. When α- or β-benzylnaphthalene is oxidised with dilute nitric acid, it is converted into the corresponding naphthyl phenyl ketone.

A mixture of α- and *β-methyl naphthyl ketone*, $C_{10}H_7 \cdot COMe$, is obtained when the reaction-product of naphthalene, acetic anhydride, and aluminium chloride is fractionated. It is a yellowish liquid boiling at about 295°, and yields naphthoic acid and sodium acetate when heated with soda-lime. A mixture of α- and β-phenyl naphthyl ketone can be obtained by treating a mixture of naphthalene and benzoic chloride with zinc or aluminium chloride (compare Merz and Kollarits, this Journal, 1873, 1035). F. S. K.

Oxidation of β-Naphthol to Orthocarboxycinnamic Acid. By E. Ehrlich and R. Benedikt (*Monatsh.*, **9**, 527—532).—When to a solution of 30 grams of β-naphthol and 30 grams of potash in 1 litre of water, 50 grams of permanganate dissolved in 1 litre of water is very slowly added, orthocarboxycinnamic acid, melting at 183—184°, is formed. Its properties have been correctly described by Gabriel and Michael (Abstr., 1878, 426), with the exception that these investigators give too low a melting point. Attempts to obtain the acetyl-derivative by heating it with acetic anhydride and sodium acetate were without success, the bibasic orthocarboxycinnamic acid being converted by this operation into phthalylacetic acid.

In oxidising β-naphthol by the above-mentioned method, a dye is formed as a bye-product. It can be separated from the accompanying β-naphthol by boiling with benzene, in which it is almost insoluble. It forms a greenish-blue solution with aqueous potash, dissolves readily in alcohol and glacial acetic acid, and is best recrystallised from the latter solvent. G. T. M.

Orthamidated Aromatic Mercaptans. By P. Jacobson (*Ber.*, **21**, 2624—2631; compare Abstr., 1887, 961).—In order to determine the constitution of α-amidonaphthyl mercaptan (*loc. cit.*), the ethenyl-compound, $C_{10}H_6{<}\overset{N}{S}{>}CMe$, was oxidised with permanganate in acid solution, when phthalic acid was obtained; the mercaptan has therefore the constitution [$NH_2 : SH = 3 : 4$].

Thiocarbamidonaphthyl mercaptan, $C_{10}H_6<^{N}_{S}>C\cdot SH$, is obtained by heating an alcoholic solution of diamidodinaphthyl sulphide,

$$NH_2\cdot C_{10}H_6\cdot S\cdot S\cdot C_{10}H_6\cdot NH_2,$$

(*loc. cit.*), with carbon bisulphide, at 110—130° for three to four hours. It crystallises from dilute alcohol in small, colourless needles which melt at above 220°. When the alkaline solution is treated with potassium ferricyanide, the *bisulphide*, $C_{10}H_6<^{N}_{S}>C\cdot S\cdot S\cdot C<^{N}_{S}>C_{10}H_6$, separates in needles.

β-Thioacetonaphthalide, $C_{12}H_{11}NS$, is prepared by fusing β-acetonaphthalide (5 parts) with phosphorus pentasulphide (3 parts), extracting the product with aqueous soda, precipitating with carbonic anhydride, and crystallising from alcohol. It crystallises in needles and in tabular crystals, and melts at 145—146°. When oxidised with potassium ferricyanide, *ethenylamido mercaptan*, $C_{10}H_6<^{N}_{S}>CMe$, is formed. This crystallises in lustrous plates, melts at 81°, and distils unchanged; the *platinochloride* forms yellow, microscopic needles.

When ethenylamidophenyl mercaptan is heated with an equal weight of phthalic anhydride and some zinc chloride at 180° to 200° for four hours, the *phthalone*, $C_6H_4<^{N}_{S}>C\cdot CH{:}C_2O_2{:}C_6H_4$, is obtained. This forms lustrous, yellow needles, melts at above 320°, and sublimes almost without change in yellow flakes. It is sparingly soluble in hot glacial acetic acid and alcohol. The *sodium sulphonate* crystallises in orange-coloured needles.

The *phthalone* of ethenyl-α-amidonaphthyl mercaptan, $C_{20}H_{11}NSO_2$, prepared by digesting the mercaptan with phthalic anhydride (0·8 part) and some zinc chloride for two hours at 190—210°, crystallises from nitrobenzene in small, yellowish-brown, pointed needles which are still more sparingly soluble than the phenyl-derivative. When heated with strong hydrochloric acid at 220—230°, ethenylamidomercaptan and phthalic acid are formed.

N. H. M.

Isomeric Naphthyl Phenyl Ketones. By O. Kegel (*Annalen*, **247**, 178—189).—Each of the naphthyl phenyl ketones prepared by the action of zinc on a mixture of benzoic chloride and naphthalene, yields a crystalline ketoxime when treated with an alcoholic solution of hydroxylamine hydrochloride at 100°.

The α-ketoxime melts at 140—142°. The β-ketoxime melts at 174—176°, and is less soluble in alcohol than its isomeride. Both compounds slowly undergo spontaneous decomposition, forming oily products. The ketones are converted into quinones when they are oxidised in acetic acid solution by chromic acid.

α-Benzoylnaphthaquinone, $C_6H_3Bz<^{CO\cdot CH}_{CO\cdot CH}>$, crystallises in yellow needles, and melts at 152°. It is freely soluble in hot alcohol, benzene, and glacial acetic acid, and resembles α-naphthaquinone in its

properties. The *quinol* is best prepared by reducing the alcoholic solution of the quinone with stannous chloride. It is soluble in hot benzene and melts at 190—191° with decomposition. The acetic derivative formed by the action of acetic chloride on the quinol crystallises in plates and melts at 154—155°.

Anilido-α-benzoylnaphthaquinone is deposited in dark-red needles when an alcoholic solution of the quinone and aniline is heated at 100° for 20 minutes, and then allowed to cool. The substance melts at 199—200°. *Paratoluido-α-benzoylnaphthaquinone* melts at 196—197°. This and the preceding compound dissolve in dilute sodium hydroxide solution containing alcohol. The dark-violet liquid turns pale-red when boiled; on acidifying the solution, *α-benzoylhydroxynaphthaquinone* is deposited in golden plates melting at 220—222°.

β-Benzoylnaphthaquinone melts at 130—132° and the quinol at 200° with decomposition. *Anilido-β-benzoylnaphthaquinone* melts at 209—210°, and is almost insoluble in alcohol.

These quinones may be regarded as α-naphthaquinone, in which benzoyl, $C_6H_5 \cdot CO$, has displaced a hydrogen in the α- and β-position respectively in that benzene nucleus which does not contain the oxygen-atoms. The α-quinone yields a benzoylphthalic acid (m. p. 128°) on oxidation, and the β-quinone appears to yield a benzoylphthalic acid melting at 150—151°. W. C. W.

Probable Orthoquinone derived from Anthraquinone. By W. H. Richardson (*Chem. News*, **58**, 54—55).—Alkaline solutions of alizarin undergo oxidation readily with the usual agents, but best of all with permanganate or bromine. On acidifying such an oxidised solution, a fawn-coloured precipitate is obtained, which by reduction is reconverted into alizarin. It, moreover, yields dyes with phenylhydrazine; and its sulphonic acid, an anilide with aniline, and its solution reacts easily with orthodiamines. The author assumes that the substance is most likely a quinone or orthodiketone corresponding with alizarin. Further experiments are in progress.

D. A. L.

Essence of Rosewood. By H. Morin (*J. Pharm.* [5], **18**, 17, from *La Nature*, 14th April, 1888).—An essential oil occurs in commerce under the name of *Essence de bois de rose femelle*, which is obtained by the distillation, in the presence of water, of the wood of *Licari kanali* (*Licaria guianensis*) an acrodiclidium of the Lauraceæ order. The essence is limpid, slightly coloured, less dense than water, burns with a smoky flame, and remains liquid at −20°. Its composition is analogous to that of camphor. Distillation of its hydrochloride yields a hydrocarbon, *licarene*, a very mobile, colourless liquid, without action on polarised light, sp. gr. 0·835 at +18°; it has the composition $C_{20}H_{16}$, and consequently is an isomeride of terebenthene.

The odour of the essence recalls that of the rose, mignonette, and orange. J. T.

Shellac. By R. Benedikt and F. Ulzer (*Monatsh.*, **9**, 579—585).—When seedlac (2 kilos.) and sodium carbonate (1 kilo.) are

boiled with water (40 litres) for some time, an oil which solidifies on cooling collects on the surface of the liquid. The wax thus obtained, weighing from 0·5 to 1·0 per cent. of the seedlac taken, has a yellowish-grey colour, and melts at 59—60°. By treatment with alcoholic potash, stearic, palmitic, and oleïc acids, together with myricyl and ceryl alcohols, may be isolated: the latter is best separated by heating with acetic anhydride, the resulting acetates being readily separated by fractional crystallisation from alcohol. The wax contains altogether about 50 per cent. of free alcohols.

The authors have proved the presence of resin wax in a sample of commercial shellac examined by them, but are at present unable to say whether or not it is a natural constituent or is simply due to colophony having been melted in with the shellac. G. T. M.

Catalpin. A Bitter Principle. By E. Claassen (*Amer. Chem. J.*, **10**, 328—330).—The fruit or bark of *Catalpa bignonioides* is extracted with alcohol, the solution evaporated, the residue dissolved in water, precipitated by lead acetate, the filtrate mixed with barium carbonate, evaporated and extracted with ether-alcohol. The solution evaporated and the residue again dissolved in water. From this solution the catalpin cannot be crystallised except by treating with animal charcoal, filtering, and washing with water, and then extracting the dried charcoal with boiling alcohol; from this solution, the principle separates in silky, acicular crystals. It is a glucoside. Its alcoholic solution is precipitated by addition of chloroform, and from its aqueous solution it is precipitated by basic lead acetate. H. B.

Quercetin. By J. Herzig (*Monatsh.*, **9**, 537—547).—Ethylquercetin is best formed by heating quercetin (12 grams) dissolved in absolute alcohol (300 c.c.) with potash (8 grams) and a corresponding quantity of ethyl iodide for four or five hours in a flask provided with a reflux condenser. The yield is from 62 to 73 per cent. of theory. Ethylquercetin has the characteristic yellow colour of quercetin-derivatives, but when heated with acetic anhydride and sodium acetate gives a colourless acetyl-derivative, crystallising from alcohol in glistening needles melting at 151—153°.

Both ethylquercetin and methylquercetin have a yellow colour, are insoluble in potash, and, therefore, contain no free hydroxyl-groups. In their forming acetyl-derivatives with change of colour, they resemble the quinones. The acetyl-derivatives are decomposed by solution in sulphuric acid: ethylquercetin and methylquercetin respectively being regenerated in theoretical quantity. The author has utilised this property to determine the molecular weight of quercetin, which he finds approaches 292 or perhaps twice that number. G. T. M.

Quercetin-derivatives. By J. Herzig (*Monatsh.*, **9**, 548—561; compare preceding Abstract).—If the molecular weight of quercetin be taken as 584, *rhamnetin* must be regarded as a dimethylquercetin. Treated with methyl iodide and potassium hydroxide, it furnishes a methylrhamnetin identical with the methylquercetin mentioned in the

previous Abstract. This compound is almost insoluble in cold alcohol, but crystallises from a hot solution in beautiful, lustrous, yellow, anhydrous needles melting at 154—155°; and on heating with acetic anhydride and sodium acetate, it yields an acetyl-derivative identical with acetylmethylquercetin. Rhamnetin is readily converted into quercetin by treatment with excess of hydriodic acid. G. T. M.

Rhinanthin. By T. L. Phipson (*Chem. News*, **58**, 99).—Leaves and stalks of *Antirhinum majus* (snap-dragon) are soaked in cold water for a few days, filtered, precipitated with lead acetate, the slight excess of lead acetate is removed from the solution by means of hydrogen sulphide, and the solution evaporated to a thin syrup. After being a few days in a warm, dry place, the syrup deposits rhombic crystals of rhinanthin, which are purified by a second crystallisation from water, and show the properties of rhinanthin obtained from other sources. The author's analyses lead to the formula $C_{64}H_{56}O_{40}$, instead of Ludwig's formula, $C_{38}H_{52}O_{40}$. D. A. L.

Strophantin. By Arnaud (*Compt. rend.*, **107**, 179—182).—The crushed seeds of *Strophantus kombé* are treated with boiling alcohol of 70° for several hours, and the solution distilled to a small bulk on a water-bath; the distillation is finished in a vacuum, care being taken that the extract remains liquid. The residue is cooled, the supernatant oil and resin separated, the liquid filtered, and heated with a small quantity of basic lead acetate and some finely powdered litharge. The liquid is again filtered, the lead removed by means of hydrogen sulphide, and the filtrate concentrated at 50° to a thick syrup, from which the strophantin slowly crystallises. The crystals may be purified by recrystallisation from boiling water; the yield is 4·5 grams per kilo.

Strophantin is a white bitter substance, which crystallises in micaceous plates, grouped round a nucleus. It readily retains water mechanically, and also forms a hydrate, which loses its water in a vacuum or in dry air. The hydrate melts below 100°, and the residual strophantin is uncrystallisable. If, however, strophantin is carefully dried in a vacuum, it may be heated at 110° without alteration. Anhydrous strophantin becomes pasty at 165°, and partially decomposes. It acts on polarised light: the rotatory power of a 2·3 per cent. aqueous solution $[\alpha]_D = +30°$. It is only slightly soluble in water, and somewhat soluble in alcohol, but is insoluble in ether, carbon bisulphide, and benzene. It is precipitated from its aqueous solutions by tannin.

Strophantin contains no nitrogen, and has a composition corresponding with the formula $C_{31}H_{48}O_{12}$. It seems to be the immediate higher homologue of ouabaïn (this vol., p. 848), which it closely resembles in properties. Perhaps these and all other cardiac poisons have a common nucleus. C. H. B.

Diosmin. By P. Spica (*Gazzetta*, **18**, 1—9).—In the author's former examination of the leaves of *Barosma crenata* and *B. betulina*

(Abstr., 1885, 1142), it was found that after the essential oil had been removed, alcohol extracted from the leaves a crystalline substance, which he called *diosmin*. The amount present in the leaves varies very much, not only with the season when they are gathered, but also with the age of the plant; at no time, however, is it large. The best way of extracting it is first to treat the leaves with light petroleum to remove the essential oil and waxy and resinous matters, then with cold alcohol of 85 per cent., which removes the chlorophyll and acid extractive substances, and finally to treat it with boiling alcohol of 80 to 85 per cent., which is the best solvent for the diosmin. It is very troublesome to purify, but this may be effected by treating the residue left on evaporation of the alcoholic solution successively with a solution of ammonium carbonate, cold alcohol, and ether, and then recrystallising repeatedly from alcohol of 80—85 per cent.

When pure, diosmin is a white, crystalline powder, consisting of very slender microscopic needles, odourless, tasteless, and insoluble in most solvents. Its best solvent is boiling alcohol of 80—85 per cent. It melts at 243—244°, with decomposition and evolution of gas. If cautiously melted on platinum foil, it emits a pleasant odour resembling that of orange peel when it begins to burn; subsequently the odour is like that of caramel. Diosmin does not reduce Fehling's solution. It dissolves in concentrated sulphuric acid, and in solutions of the alkalis with yellow coloration, but at the same time undergoes alteration.

When diosmin is boiled for some time in a reflux apparatus with dilute hydrochloric acid, or with sulphuric acid of 3—4 per cent., it is resolved into a crystalline compound, sparingly soluble in water, and a substance easily soluble in water; the latter has a feeble dextrorotatory power, and is capable of reducing Fehling's solution. The crystalline substance is moderately soluble in alcohol, and still better in a mixture of alcohol and benzene. It forms minute, orange-yellow needles, for which no definite melting point could be obtained, but it appears to be somewhere about 145°.

The results obtained by the analyses of diosmin dried in a vacuum over sulphuric acid are, C = 53·43, H = 6·17 (mean of three analyses), which closely agree with the numbers obtained by Paternò and Briosi for hesperidin, namely, 53·44 and 5·92; but E. Hoffman, taking the formula as $C_{22}H_{26}O_{16}$ (C = 54·77 and H = 5·39), considers that the sample analysed by Paternò and Briosi was incompletely dried, and that a temperature of 150° is necessary to remove all the water.

On heating diosmin in a current of air at 150—160°, it lost 4·2 per cent. of water, and on analysis then gave numbers corresponding with C = 57·77 and H = 6·00. The author is making a careful comparison of diosmin with hesperidin, in order to ascertain whether they are identical or not. C. E. G.

Constitution of Quassin. By V. Oliveri (*Gazzetta*, **17**, 570—577).—Of the derivatives of quassin, quassic acid seems to be the compound which is best adapted for investigation, in order to obtain an insight into the constitution of quassin. In order to prepare the

acid, it is advisable to operate on small quantities of quassin at a time (5 grams); this is heated at 100° for one hour in a sealed tube with hydrochloric acid (40 c.c.) diluted with its own volume of water. The solution is separated from resinous matter by filtration, and precipitated with water. The precipitate, after being washed, is crystallised several times from alcohol, by which means the unaltered quassin is easily separated, as the quassic acid is comparatively insoluble in cold alcohol.

Quassic acid, $C_{30}H_{38}O_{18}$, crystallises in small, colourless, monoclinic prisms, containing 1 mol. H_2O, which they lose at 100°. It melts with decomposition about 244—245°, is soluble in boiling alcohol, but only sparingly in cold alcohol and ether. It dissolves in ammonia, and in solutions of alkaline carbonates with a yellow colour, more or less inclined to red. Its ammoniacal solution gives precipitates with mercuric chloride, lead acetate, copper sulphate, and ferric chloride, and reduces silver nitrate; its aqueous solution is coloured greenish-yellow by ferric chloride.

In a previous communication (Abstr., 1885, 907), published by the author in conjunction with Denaro, quassin was regarded as the dimethyl salt of quassic acid. This the author has now confirmed by proving that methyl chloride is formed by the action of hydrochloric acid on quassin, and that quassic acid is bibasic. For this purpose, several of its salts were prepared and analysed. *Barium quassate*, $C_{30}H_{36}O_{10}Ba,7H_2O$, forms a yellowish-red, crystalline mass. The *lead salt*, $C_{30}H_{36}O_{10}Pb,6H_2O$, is thrown down as a yellow precipitate, on adding acetate of lead to a neutral solution of ammonium quassate; *ferric quassate*, $(C_{30}H_{36}O_{10})_3Fe_2$, is a brownish-green amorphous precipitate.

On adding a solution of hydroxylamine hydrochloride (6 grams) to quassic acid (10 grams), dissolved in sodium carbonate, an abundant precipitate of the *dioxime of quassic acid*, $C_{28}H_{36}O_8(C{:}NOH)_2$, is thrown down after a little time. It may be purified by crystallisation from alcohol diluted with its own volume of water, when it is obtained in yellowish rectangular prisms; it melts at 228—230°, and is at the same time completely decomposed.

When quassin is heated with concentrated hydriodic acid (sp. gr. 1·70) and some amorphous phosphorus for 20 hours, at a temperature of 150°, gradually increased to 280°, a reducing action takes place, which varies with the temperature; at 150°, methyl iodide is produced, whilst at the higher temperature resinous products and a mixture of hydrocarbons are obtained. The latter, separated from the filtered and neutralised solution by distillation with steam, appeared as a yellowish, oily liquid, which on being submitted to fractional distillation yielded three definite hydrocarbons. The first, boiling at 188—195°, was found to have the composition of β-durene, $C_{10}H_{16}$, and by treatment with bromine yielded a brominated derivative crystallising in colourless needles melting at about 200°. The second fraction, boiling at 220—240°, had the composition $C_{14}H_{16}$, and gave a brominated derivative, which crystallised from alcohol in colourless needles melting at about 150°, and subliming at a lower temperature. The residue which did not come over at 240°, contained anthracene.

The author considers it proved that quassin contains four hydroxyl groups, OH, two carboxymethyl groups, COOMe, and two ketonic groups, CO. From other results obtained, which he hopes soon to publish, it would appear that quassin is an anthraquinone-derivative.

C. E. G.

Jecorin in the Animal Body. By D. Baldi (*Chem. Centr.*, 1888, 978, from *Arch. physiol.*, 1887, Suppl., 100).—The author has obtained jecorin from rabbit's liver, ox spleen, human brain, and horse's muscle, and it appears to constantly accompany lecithin. Next to the liver, the spleen gave the highest yield. Jecorin reduces Fehling's solution, although its reducing power appears to vary with the source from which it is obtained. J. W. L.

Dye from Seaweed. By F. Nettlefold (*Chem. News*, **58**, 15).—By nitrating alginic acid, a light-yellow nitro-product is obtained insoluble in water, but soluble in alkalis to a brown solution. The alkaline, best ammoniacal, solution dyes unmordanted cotton a fine Bismark-brown, fast to soap, excelling many aniline colours, and equalling chrysoïdine; the depth of shade is considerable and can be worked to a great intensity. Neither the colour nor the fastness is increased by mordanting with alumina or potassium antimony tartrate. This dye has little affinity for wool, mordanted or unmordanted. In acid solutions it does not dye. D. A. L.

Pyridine and Piperidine Bases formed from Acetone and Aldehyde-ammonia. By E. Dürkopf (*Ber.*, **21**, 2713—2718).—When aldehyde-ammonia and acetone are heated together at 200° for 9 to 10 hours, a secondary and tertiary base are obtained, and can be separated by treating the mixture of their hydrochlorides with sodium nitrite. The tertiary base is symmetrical trimethylpyridine; it boils at 167—168°, and yields a copellidine (see below) when reduced with sodium and alcohol. The aurochloride melts at 106° with previous softening (compare Mohler, *Ber.*, **21**, 1011). The secondary base is a copellidine, $C_8H_{17}N$. The platinochloride decomposes at 242—244° (compare Jaeckle, this vol., p. 1103). F. S. K.

Action of Ethyl Acetoacetate on Hexamethylenetetramine. By P. Griess and G. Harrow (*Ber.*, **21**, 2740—2743).—When a mixture of ethyl acetoacetate (20 grams) and hexamethylenetetramine (4 grams) is heated at 170°, a small quantity of ethyl lutidinedicarboxylate is formed, but the chief product is a yellow, amorphous substance; if, however, chloride of zinc (20 grams) is added, only small quantities of the amorphous substance are formed, and considerable quantities of ethyl lutidinedicarboxylate (compare Engelmann, Abstr., 1886, 259) and ethyl hydrolutidinedicarboxylate are produced.

Ethyl hydrolutidinedicarboxylate, $C_5NH_3Me_2(COOEt)_2$, separates almost completely when the product of the reaction is mixed with hot water, the cold solution filtered, and the residue extracted with boiling alcohol. It crystallises in anhydrous needles or in greenish-yellow, quadratic plates, melts at 170° with previous softening, and is readily

soluble in chloroform, but only very sparingly in cold alcohol and ether, and almost insoluble in boiling water. It has a neutral reaction, and distils with considerable decomposition. When treated with nitrous acid in alcoholic solution, or when dissolved in hot, dilute hydrochloric acid, it is converted into diethyl lutidinedicarboxylate, melting at 72°, but in the latter reaction, as a rule, a considerable quantity of monethyl lutidinedicarboxylate, $COOH{\cdot}C_5NHMe_2{\cdot}COOEt$ (compare Weiss, Abstr., 1886, 719) is also formed. F. S. K.

Constitution of the Parvoline obtained from Propionaldehyde-ammonia and Paraldehyde. By E. Dürkopf and M. Schlaugk (*Ber.*, **21**, 2707—2708).—The pyridinecarboxylic acid which the authors obtained by oxidising parvoline (compare this vol., p. 607) is identical with carbodinicotinic acid (Weber, Abstr., 1887, 1117). It melts at 315° as the average of several observations, crystallises with 2 mols. H_2O, and is converted into dinicotinic acid when heated at 150°. F. S. K.

Piperidine Dyes. By B. Lachowicz (*Monatsh.*, **9**, 505—509).—When piperidine is added to quinone dissolved in alcohol, a reddish-brown solution is obtained, from which a dye, *dipiperylquinone*, $C_6H_2O_2(C_5H_{10}N)_2$, separates in thick, reddish-violet prisms. It melts at 178°, is insoluble in water, but dissolves in alcohol and dilute hydrochloric acid. Its mode of formation is shown by the equation $3C_6H_4O_2 + 2C_5H_{11}N = C_6H_2O_2(C_5H_{10}N)_2 + 2C_6H_4(OH)_2$. Digestion with strong hydrochloric acid converts it into dihydroxyquinone and piperidine hydrochloride.

Dipiperylquinone is analogous in constitution to the compound obtained by Mylius (Abstr., 1885, 804) from the interaction of dimethylamine and quinone, but the mode of formation is altogether different; in the latter case, oxidation is brought about by atmospheric oxygen, whereas in the production of dipiperylquinone by the above method oxidation takes place at the expense of a portion of the quinone present. Dipiperylquinone may, however, be readily obtained by a process identical with that used by Mylius, namely, by exposing mixtures of quinol and piperidine to the action of atmospheric oxygen. Piperidine forms dyes with toluquinone, naphthaquinone, and phenanthraquinone, but the reaction does not take place nearly so readily as with quinone, nor has the author yet been able to obtain the compounds in a pure condition.

G. T. M.

Furfurethenepyridine. By E. Merck (*Ber.*, **21**, 2709—2712).—*Furfurethenepyridine*, $\begin{matrix}CH{\cdot}CH\\CH{\cdot}O\end{matrix}\!\!>\!C{\cdot}CH{:}CH{\cdot}C_5NH_4$, is formed when a mixture of furfuraldehyde and α-picoline is heated at 160—170° with a small quantity of zinc chloride. The yield is about 10 per cent. of the picoline employed. It crystallises from water in white needles, quickly blackens on exposure to the air, and is readily soluble in alcohol and ether, but almost insoluble in water. The freshly distilled base has a slight but characteristic odour, and melts at 51—53°. The *mercurochloride*, $C_{11}H_9NO,HHgCl_3$, crystallises in yellow needles,

and melts at 133°. The *platinochloride*, $(C_{11}H_9NO)_2,H_2PtCl_6 + 2H_2O$, crystallises in microscopic, reddish-yellow needles melting at 155° with decomposition. The *picrate*, $C_{11}H_9NO,C_6H_3N_3O_7$, crystallises in yellow needles and melts at 185—190°. The *aurochloride* is reddish-brown.

Furfurethanepiperidine, $\begin{matrix}CH\cdot CH\\ CH\cdot O\end{matrix}\!\!>\!C\cdot CH_2\cdot CH_2\cdot C_5H_{10}N$, obtained by reducing the preceding compound with sodium and alcohol, is a strongly alkaline liquid with a marked piperidine-like odour. It boils at 245—247°. The *hydrochloride*, $C_{11}H_{17}NO,HCl$, melting at 145—148°, the *hydrobromide*, melting at 133—135°, and the *hydriodide*, melting at 119—121°, are crystalline. The base is a powerful poison.

F. S. K.

Alkaloïds from Cod-liver Oil. By A. Gautier and L. Mourgues (*Compt. rend.*, **107**, 110—112, and 254—257).—Pale or fawn-coloured oil from Norway or Sweden is treated with its own volume of alcohol of 33° containing 4 grams of oxalic acid per litre. The alcohol is separated, saturated almost exactly with calcium oxide, filtered, and distilled in a vacuum at 45°. The product is digested with calcium carbonate, neutralised with lime-water, evaporated to dryness in a vacuum, and the residue extracted with alcohol of 90°. The alkaloïds are purified in the usual manner; the yield is 0·35 to 0·5 gram per kilo.

The fraction boiling at 87—90° consists of butylamine; at 96—98°, amylamine; just below 100°, hexylamine; at 198—200°, a new base, *hydrolutidine*. The non-volatile residue contains a new base, *aselline*, the hydrochloride of which is insoluble, and a second, *morrhuine*, which forms a soluble, crystallisable platinochloride. Cod-liver oil also contains a small quantity of lecithin and a peculiar crystallisable nitrogenous acid, *gaduinic acid*.

Butylamine has distinct toxic properties, and in small doses increases the activity of the functions of the skin and the kidneys. Larger quantities produce fatigue, stupor, and vomiting. The isoamylamine, which constitutes about one-third of the total volatile bases, is identical with the isoamylamine obtained by the action of potassium hydroxide on isoamylcarbimide. It exerts a powerful physiological action, 4 mgrms. of the hydrochloride having killed a greenfinch in three minutes. In small doses, it excites reflex action, and the urinary secretions; in large doses, it produces general tremor, well-marked convulsions, and death. Hexylamine, which is present in very much lower proportion, produces similar effects but is much less powerfully toxic.

The fraction of the bases which boils at 198—200° under a pressure of 770 mm. consists of *dihydrolutidine*, $C_7H_{11}N$, a colourless, oily, strongly alkaline and caustic liquid; it boils at 199° under normal pressure, and its vapour-density at 299° = 3·3. When exposed to air it absorbs carbonic anhydride, becomes viscous, and darkens in colour. The hydrochloride crystallises in confused needles which seem to belong to the rhombic system. The nitrate, like the nitrates of hydropyridic bases, reduces a solution of silver nitrate. The sulphate crystallises in stellate groups of deliquescent needles. All these salts

have a bitter taste. The platinochloride is only slightly soluble, and crystallises from a hot solution in canary-yellow lozenge-shaped lamellæ. When boiled with a large quantity of water, it loses hydrogen chloride, and is converted into a modified salt, $(C_7H_{11}NCl)_2,PtCl_2$, which is much less soluble than the original compound, and forms confused crystals of a paler colour. The aurochloride crystallises in needles or plates, and is somewhat stable even when heated.

The methiodide forms readily at the ordinary temperature with development of heat, and crystallises in colourless needles which dissolve in water or alcohol, and have a disagreable and somewhat nauseous odour. With potassium hydroxide, it yields dihydromethyllutidine, a colourless, strongly alkaline aromatic oil. When dihydrolutidine is boiled with potassium permanganate, an aromatic odour resembling that of coumarin is observed, and this points to the intermediate formation of a compound containing the aldehyde-group. Oxidation is completed in a sealed tube at 100°, and the product is methylpyridinecarboxylic acid, $C_5NH_3Me{\cdot}COOH$. It follows that the dihydrolutidine is dihydrodimethylpyridine, $C_5NH_5Me_2$.

Dihydrolutidine is moderately poisonous, and in small doses reduces the general sensibility. Larger doses produce tremor, especially of the head; depression, with alternating periods of intense excitement and paralysis of the lower members, ending in death.

C. H. B.

Narcotine. (Part II.) By W. Roser (*Annalen*, **247**, 167—177).—Methyl iodide unites with narcotine, forming narcotine methiodide, an oily liquid. Narcotine metho-chloride and the platinochloride are crystalline. On the addition of sodium hydroxide to narcotine metho-chloride, narcotine methyl hydroxide is precipitated, but this compound soon changes into pseudonarceïne, $C_{23}H_{27}NO_8$, and the transformation is greatly facilitated by exposure to steam. A careful examination of pseudonarceïne and its derivatives brings the author to the conclusion that this substance is identical with narceïne. The formation of pseudonarceïne from narcotine methyl hydroxide is represented by the following equation: $C_{22}H_{23}NO_7,CH_3{\cdot}OH + 3H_2O = C_{23}H_{27}NO_8,3H_2O$.

W. C. W.

Conversion of Hyoscyamine into Atropine by Bases. Action of Mass. By W. Will and G Bredig (*Ber.*, **21**, 2777—2797; compare Will, this vol., p. 855).—The authors investigated the action of soda on a solution of hyoscyamine containing 6·6667 grams in 100 c.c., and on a solution of twice this concentration. The results, which are given in tabular and diagrammatic form, show that in the same time the rotatory power of the more concentrated solution is diminished twice as much as that of the more dilute solution. The action of the soda is, therefore, a catalytic action.

Tables are also given showing that to produce a certain decrease in the rotatory power of a solution containing a given quantity of hyoscyamine, a definite volume of normal soda takes four times as long as the same volume of a solution containing four times as much soda. The results obtained with dimethylamine show that the action of this base is quite similar to that of soda.

The decrease in the rotatory power of solutions containing the same quantity of hyoscyamine, under the influence of soda, potash, tetramethylammonium hydroxide, dimethylamine, and ammonia, of different concentrations, is given in tabular form, and shows that ammonia and dimethylamine act much more slowly than the other three bases.

From the observed final rotatory power of solutions of hyoscyamine under the influence of soda, potash, and tetramethylammonium hydroxide, based on the assumption that the hyoscyamine has been completely converted into atropine, the specific rotatory power of the latter is $[\alpha]_D = -1{\cdot}89$.

When the conversion of hyoscyamine into atropine is almost complete, a small quantity of the latter is decomposed into tropine and tropic acid; owing to this secondary reaction the relative values of the velocity constants are increased in the case of soda, potash, and tetramethylammonium hydroxide, and decreased in the case of ammonia and dimethylamine.

The authors intend to try and measure the action constants of bases by a more complete study of this phenomenon. Sodium carbonate converts hyoscyamine into atropine, but ammonia has the slowest action of all the bases experimented with. F. S. K.

Tetanine and Mytilotoxine. By L. Brieger (*Chem. Centr.*, 1888, 1007, from *Arch. pathol. Anat.*, **112**, 549—551).—The hydrochlorides of these bases decompose gradually, and lose their toxic properties. The product of decomposition of the tetanine hydrochloride gave a platinochloride of the formula $C_6H_{13}NO_2,H_2PtCl_6$. Mytilotoxine is best preserved in the form of aurochloride.

J. W. L.

Anagyrine. By E. Hardy and N. Gallois (*Compt. rend.*, **107**, 247—249).—The authors discovered anagyrine in 1885 (*Compt. rend. Soc. Biol.*, 1885, 391) two years before Reale's description of the alkaloïd (*Gazzetta*, 1887, 385).

The seeds of *Anagyris fœtida* are macerated with cold water, treated with basic lead acetate, the lead removed by hydrogen sulphide, and the filtrate concentrated and mixed with mercuric chloride, which precipitates the anagyrine. The precipitate is decomposed by hydrogen sulphide, and the concentrated filtrate saturated with sodium carbonate and agitated with chloroform, the chloroform solution being afterwards agitated with hydrochloric acid, from which the hydrochloride crystallises on evaporation.

Anagyrine, $C_{14}H_{18}N_2O_2$, is a yellowish, amorphous substance which dissolves in water, alcohol, or ether, and softens and becomes viscous when exposed to the air. Its solutions give a white precipitate with potassium mercuriodide, a brown precipitate with iodine solution, and the usual general reactions for alkaloïds. With acids, it forms salts which, contrary to the statement of Reale, crystallise readily.

Anagyrine hydrochloride, $C_{14}H_{18}N_2O_2,HCl + 4H_2O$, forms white, rectangular tables belonging to the rhombic system, does not alter when exposed to air, and dissolves very readily in water or chloroform,

but is less soluble in alcohol, and only slightly soluble in ether. The crystals become anhydrous at 125°. The rotatory power of the hydrochloride is $[\alpha]_D = -114°$. The *aurochloride*,

$$C_{14}H_{18}N_2O_2,HAuCl_4,$$

forms at first a yellow, amorphous precipitate, but this rapidly becomes crystalline. The *platinochloride*, $C_{14}H_{18}N_2O_2,H_2PtCl_6$, separates in crystalline tufts. All these salts were analysed, and the composition of the alkaloïd was thus definitely determined.

Anagyrine has a moderate toxic action. In warm-blooded animals it produces vomiting, coldness, and tremor, slackening of the respiratory movements, and finally stoppage of respiration and circulation. When administered to a frog, the abolition of muscular movements is very remarkable; the motions of the heart persist long after all other movements have ceased. C. H. B.

Constitution of Spongin. By P. Zalocostas (*Compt. rend.*, **107**, 252—254).—The organic matter of sponges, previously treated with hydrochloric acid and with benzene, was heated in sealed tubes with a solution of barium hydroxide as in Schutzenberger's experiments with proteïds. The products are ammonia, carbonic anhydride, acetic acid, oxalic acid, and a mixture of solid nitrogenous bases, namely, leucine, butalanine, glycalanine, traces of tyrosin, and a hydroproteic acid or hydrate of leuceine. The ammoniacal nitrogen is equivalent to one-fourth of the total nitrogen, and 2 mols. of ammonia are formed for each molecule of carbonic anhydride. The number of molecules of water assimilated is equivalent to the number of atoms of nitrogen in the original compound. In the solid products, the ratio of carbon-atoms to hydrogen-atoms is 1 : 2, and that of nitrogen to oxygen 1 : 2·66. It follows from these results that spongin is closely related to the collagenous proteïds. C. H. B.

Peptonic Fermentation of Meat. By V. Marcano (*Compt. rend.*, **107**, 117—119).—If chopped flesh is mixed with the juice and tissues of the *agave* or American aloe, the solution of the fibrin is complete in five to six hours, and the yield of peptone is about 20 per cent. (Compare Abstr., 1885, 181.) If the chopped and squeezed flesh is mixed with the tissues and juice of the plant without addition of water and kept at 35°, it swells up, but little peptone is formed. The magma thus produced is mixed with water and heated to 70°, when it liquefies immediately and yields a highly concentrated solution of peptone. It would seem that a pepsin is formed and unites with the fibrin, producing a compound which is insoluble at 35° but dissolves readily in water at 70°.

Equal quantities of flesh were mixed with fresh agave tissue and with some of the same tissue after boiling with water. The formation of peptone was much greater in the first case, and hence it follows that the peptonising power resides mainly in the living vegetable cell.

This process is employed successfully on a large scale for the manufacture of peptone. C. H. B.

Physiological Chemistry.

Feeding of Calves and Pigs. By N. J. Fjord (*Bied. Centr.*, 1888, 590—601).—Experiments made with calves and pigs on the relative value of skim-milk obtained by centrifugal separators and by the Holstein tub method, the former containing 0·15 per cent. of fat and the latter 0·6 per cent., showed that the latter kind gave a slightly larger increase in weight in a given time, but that the money value of this increase was only about one-fifth of that of the butter fat sacrificed.

Some experiments with pigs on the relative feeding values of corn, skim-milk, and whey, and on other points, led to the following conclusions:—

1. The opinion that pigs make better use of their food when it is largely diluted with water was not confirmed; the differences in increase obtained being either unimportant or to the disadvantage of dilution. 2. Although confining pigs produced more increase than allowing them to run in the sty-yard, yet the author thinks it may cause disease. 3. 12 parts by weight of whey, 6 parts of skim-milk, 1 part of bruised barley, and 1 part of bruised rye are of approximately equal feeding value.

These conclusions were deduced from the average increase in weight in equal times. In the experiments for conclusion 3, on slaughtering the pigs the amount of offal and the thickness of the fat were determined, and the pigs were classified by experts according to their quality, and the results bore out fairly well the statement given above.

H. Nathorst, remarking on the above experiments, states that the equivalency of the foods mentioned is not of universal application (as Fjord too observes), for the substitution of one food for another may materially alter the albuminoïd ratio of the diet. H. H. R.

Influence of Fodder on the Production of Lean and Fat in Pigs. By Henry (*Bied. Centr.*, 1888, 606—609).—Six pigs of 100 days old, and from the same litter, were selected and divided into two equal lots. Set I received a diet of 1 part by weight of dried blood, 6 parts of bran, and 14 parts of skim-milk. Set II received an unlimited amount of maize meal. The albuminoïd ratio of the diet of set I was 1 : 2, and of that of set 2 was 1 : 7 or 8. At the end of 136 days from the commencement of the experiment, the pigs were slaughtered and the weights of different parts were determined. The weights obtained for set I in every case, except as regards fat, exceeded those for set II; the live weights were 19 per cent. greater for set I than for set II, the carcases 21, the kidneys 42, the spleens 33, the livers 32, the blood 59, the hair and skins 36, the large muscles of the back (*Ilia spinalis*) 64, the two muscles of the body cavity (*Psoas magnus*) 38, and the bones 23 per cent. greater. In set I, 38 per cent. of the bodies, excluding bones, was fat; in set II

46 per cent. of fat was present. The strength of the thigh bones was determined by a specially constructed machine, and was found to be 62 per cent. greater for set I than for set II. From these results the author infers that by varying the feeding, fat or lean can be produced at will, and also that feeding exclusively with maize or other foods over rich in carbohydrates affects the whole organism unfavourably, and so may be dangerous if the animals are used for breeding.

H. H. R.

Cutaneous Excretion of Albumin by the Horse. By A. Leclerc (*Compt. rend.*, **107**, 123—126).—The white secretion frequently observed on a horse which has perspired, yields an opalescent solution, which always contains albumin, alkaline chlorides, ammonium salts, urea, and one or more nitrogenous organic compounds. On four consecutive days, a horse which perspired freely excreted 10·308, 5·558, 4·237, and 5·596 grams of albumin respectively. Another horse, which perspired much less readily, excreted on four consecutive days 8·605, 2·701, 2·690, and 4·114 grams of albumin. It is evident that the loss of albuminoïd and other nitrogen in this manner must be taken into account in all considerations of the circulation of nitrogen.

The dried perspiration forms a white deposit which has usually been regarded as epithelial *débris*. The latter, however, is present in very small proportion, and the substance consists mainly of salts and albumin which is still completely soluble in water.

C. H. B.

Mean Composition of Normal Urine. By Yvon and Berlioz (*Lancet*, **2**, 1888, 629, from *Rev. Méd.*, **8**, 713—718).—A series of tables of the analysis of normal urine are given. The observations are very numerous, and were made on healthy adults, male and female. The present results are contrasted with those of other authors, and in each case the maxima and minima as well as the means are given. The latter are summarised thus:—

	Male.	Female.
Volume per diem	1360 c.c.	1100 c.c.
Specific gravity	1·0225	1·0215
Urea (per litre)	21·5 gram	19·0 gram
„ (per diem).....	26·5 „	20·5 „
Uric acid (per litre)	0·5 „	0·55 „
„ (per diem).....	0·6 „	0·57 „
Phosphoric acid (per litre).	2·5 „	2·4 „
„ (per diem)	3·2 „	2·6 „

Thus, with the exception of uric acid, the amounts are higher on each head among males than among females; but with uric acid, the quantities eliminated in the 24 hours are almost precisely the same for the two sexes. The authors desire to correct, as resulting from these observations, the proportionate quantities of urea and uric acid given in their *Manual of Urinary Analysis*, which should be as 40 : 1 instead of 30 : 1, and of urea and phosphoric acid, which should be as 8 : 1 instead of 10 : 1.

W. D. H.

Secretion of Urine when Pressure is exerted on the Urinary Canals. By R. Lèpine and E. Porteret (*Compt. rend.*, **107**, 74—77).—The experiments were made with dogs. A canula was placed in each ureter, and from one of these the urine was allowed to escape freely, the other being attached to an india-rubber tube. In the second case, the urine escaped from the free end of the tube, and by raising this, a back pressure was produced by the accumulation of urine in the tube, the pressure being measured of course by the height of the column of liquid. By operating in this manner, it was possible to collect and analyse the urine secreted at the same time by each kidney, the one being under pressure whilst the other was under normal conditions.

The quantity of urine secreted under pressure varies greatly in different animals. In some cases, the secretion is almost stopped, whilst in others it is only slightly reduced. In these experiments, the secretion was accelerated by the intra-venous injection of a 5 per cent. solution of sodium chloride. The quantity of urine secreted has no relation to the pressure. Under feeble pressure; it would seem that the quantity of urea diminishes in somewhat greater proportion than the volume of urine, whilst with a pressure of 45 cm. the volume of urine diminishes to a much greater extent than the quantity of urea. As a rule, the inorganic salts vary in the same ratio as the volume of urine, but under feeble pressure the ratio of chlorides is higher than that of other salts, whilst under higher pressure the reverse is the case. The proportion of phosphates also diminishes under pressure. When sugar is injected into the veins, the quantity in the urine varies directly as the volume of the latter. The urine excreted under pressure is really the resultant of a double process of secretion and resorption. C. H. B.

Paralactic Acid in the Urine of Soldiers after a Forced March. By G. Colasanti and R. Moscatelli (*Gazzetta*, **17**, 548—557).—Although it has long been known that the ordinary optically inactive lactic acid occurs in urine both normal and pathological, the presence of paralactic acid, the other ethylidenelactic acid, in normal urine has never been decisively proved; it has, however, been stated to exist in some pathological urines. The author has operated on comparatively large quantities of fresh urine collected after forced marches of 20 to 25 kilometres, and has conclusively proved the presence of paralactic acid in it. The urine, as soon as collected, was evaporated on the water-bath to a syrupy consistence, and the acid converted into the zinc salt by treatment with zinc carbonate in the way recommended by Salkowski. The crude zinc salt was repeatedly washed with absolute alcohol, to remove a brown resinous substance, and then repeatedly crystallised. By spontaneous evaporation, it was obtained as a mass of colourless, brilliant, microscopic, prismatic crystals of the composition of zinc paralactate,

$$C_6H_{10}ZnO_6 + 2H_2O,$$

agreeing in all its properties with the salt described by Wislicenus.

This proves beyond doubt that the acid which Du Bois Reymond

has shown to be produced in muscle when in activity enters into the circulation; a part is oxidised to carbonic anhydride and water, as Spiro has recently proved experimentally, and a part passes through the kidneys unaltered, and is excreted with the urine.

C. E. G.

Behaviour of Urine after Ingestion of Naphthalene. By Edlefsen (*Chem. Centr.*, 1888, 1007, from *Centr. Klin. Med.*, **9**, Beil., 90—92).—Fresh urine after the use of naphthalene, gives a blue flourescence when treated with ammonia or soda. When treated with bleaching powder and hydrochloric acid, it gives the red β-naphthaquinone reaction. Fischer's reaction with diazoamidobenzene also shows the presence of β-naphthol. The urine becomes cherry-red on standing for several days with acetic acid. Under these latter circumstances, the phenol test may also be successfully applied.

J. W. L.

Excretion of Uric Acid. By A. Haig (*J. Physiol.*, **8**, 211—217, *Medico-Chirurg. Trans.*, **71**, 125—138, and 283—295).—The administration of acids diminishes the relative amount of uric acid excreted, and that of alkalis increases it: as an instance, in one case the normal proportion of uric acid to urea was 1 : 35. After a few doses of 4 grams of citric acid, the relation was 1 : 41; after similar doses of potassium citrate, it was 1 : 28. In these cases, there is not only a relative, but also an absolute diminution and increase in the uric acid excreted. The excretion of uric acid is much affected by the digestion of food, and is three times as much during the "alkaline tide" as at other periods: a large part of this increased secretion must be regarded as a washing out of the uric acid accumulated in the liver and spleen in the "acid tide" periods between meals, or during sleep, and not as entirely due to increased formation of uric acid during digestion. Certain peculiar forms of headache most marked during the strongest "alkaline tide" as during the digestion of breakfast, are regarded as being due to the increased amount of uric acid in the circulation during that period. Such headaches may be cured by a dose of acid.

Salicylic acid, however, forms an important exception to this rule, for while it increases urinary activity it does not in any way diminish the excretion of uric acid; moreover, acids given while salicylates are present in the circulation have no longer the power of diminishing the excretion of uric acid; excessive excretion of uric acid under salicylates is not accompanied by any headache. Benzoates do not act in the same way, probably because hippuric acid which they form is less soluble than salicyluric acid. Both uric and salicyluric acids are present in the urine passed under the influence of salicylates; this is probably due to the salicylate acting on the uric acid in the blood, but not on that secreted by the kidney itself. In these experiments, Haycraft's method of estimating uric acid was employed. Salicyluric acid does not give the reactions on which this process depends.

A large part of the value of salicylates in uric acid diseases is due to their preventing acids from causing retention of uric acid. Some

drugs have the opposite action, and cause retention of uric acid. Lead, iron, and lithia are instances of these. The action of lead in precipitating an attack of gout is well known. Iron also causes relapses in gout and does harm in epilepsy and uric acid headache. On the other hand, salicylates prevent gout, the peculiar headache in question, and also epilepsy. This and certain other facts not chemical in nature seem to place epilepsy in the same category as gout, and it is considered probable that epilepsy is really an uric acid disease, the poison (uric acid) acting on the nerve-centres.

W. D. H.

Lactic Acid in the Urine of Cold-blooded Animals after Extirpation of the Liver. By E. NEBELTHAU (*Zeit. Biol.*, **25**, 123—136).—Minkowski (*Arch. Exp. Path. Pharm.*, **21**, 41) has shown that lactic acid is present in the urine of geese from which the liver has been removed, and absent in the urine of healthy geese. Marcuse has investigated the question whether the same holds true for the cold-blooded animals (*Pflüger's Archiv*, **39**, 425); he found in the case of frogs that it did. He used as a test for lactic acid Uffelmann's colour reaction with ferric chloride (*Zeit. klin. Med.*, **8**, 392); he also found that the calcium salt prepared from the acid had a percentage of water of crystallisation corresponding with that of calcium lactate. Marcuse's experiments not being considered satisfactory, from the small quantity of material he worked with, and Uffelmann's test being not wholly characteristic of lactic acid, the present research was undertaken. Preliminary experiments with tortoises proved negative, as these animals secreted no urine after the operation. Frogs were then taken. Some hundreds were kept in a tank, in water; the increase in the volume of the water was taken as being due to the urine of these animals. The urine thus took several weeks to collect, and putrefaction was prevented by the use of a small quantity of corrosive sublimate. The liver was then removed, and the urine similarly collected; by this method many litres of frog's urine was obtained.

The following are the chief analytical facts ascertained with regard to the urine before and after the operation:—

	Before operation.	After operation.
Specific gravity	1·0015	1·0025
Reaction	faintly acid	less acid
Total solids	0·1062 per cent.	0·140 per cent.
Ammonia	0·0054 „	0·0122 „
Chlorides, Sulphates, Phosphates	present	present
Uric acid	absent	absent
Urea	present	absent
Lactic acid	absent	absent

The method adopted in the case of lactic acid was to attempt to prepare zinc lactate; none was obtainable in either case. The experiment was repeated using a different method of collecting the urine;

this time it was simply squeezed several times daily from the bladder of the animals. In this way, nearly 8 litres were obtained (by the previous method only 3 litres were obtained between the operation and the death of the animals). From this quantity collected after the operation of removal of the liver, 0·1279 gram of a zinc salt was obtained, which in two of its properties resembled paralactate of zinc, namely, in the yellow colour it gave with ferric chloride, and in the lævorotatory action of its solution, on polarised light. The quantity obtained was insufficient to make trustworthy estimations of water of crystallisation, and the crystalline form of the substance gave no very conclusive evidence. W. D. H.

Thiocyanic Acid in the Animal Organism. By J. Bruylants (*J. Pharm.* [5], **18**, 104—107, 153—156). The identification of thiocyanic acid was based on the following facts: When an aqueous solution of the above compound is treated with an excess of hydrochloric acid and with ether, the thiocyanic acid passes completely into the ether without decomposition, provided not too much be present. When a thiocyanate is distilled with an excess of a strong mineral acid, most of the thiocyanic acid passes over with the first portions of the distillate. In saliva from healthy subjects, the amount found corresponded to 0·0963 gram of ammonium thiocyanate per litre. Human urine averaged 0·0027 gram per litre, about one-tenth of the amount admitted by Gscheidlen, and one-fortieth of the average proposed by Munck. There appears to be no definite relation between the amount found in saliva and the amount found in urine. Horse's urine contains a little more than human urine. Cow's urine averages 0·0042 gram per litre. Defibrinated cow's blood contained 0·00075 gram per litre. Serum from cow's blood gave 0·0009 gram; and dried albumin contained 0·0095 gram per kilo. Cow's bile contained 0·01 gram per litre. Three samples of cow's milk gave respectively 0·0008, 0·0024, 0·0016 gram per litre. The foregoing results show that thiocyanic acid is found not only in saliva, but in most, if not all, physiological and pathological fluids. It is difficult to think that this compound is formed exclusively in the saliva, since in some individuals this secretion has only contained traces, whilst the urine contained relatively large amounts. J. T.

Blood Changes in Disease. P. W. Latham (*Lancet*, **2**, 1888, 751—756).—This address constituted the Harveian oration for the present year, and will be found to contain many suggestions of interest both to the chemist and biologist. Among other points, it is shown how it is possible to obtain certain of the poisonous alkaloïds or ptomaïnes, on the supposition that the author's theory of the constitution of albumin is correct. (Abstr., 1886, 635.) W. D. H.

Pernicious Anæmia. By W. Hunter (*Lancet*, **2**, 1888, 555—559, 608—611, 654—648). In an elaborate research, clinical, microscopical, and chemical, into the pathology of the obscure disease known as pernicious anæmia, the following conclusions are drawn:—The essential pathological feature is an excessive destruction of the microscopic

elements of the blood, not an insufficient formation of these. The most constant change is a large excess of iron in the liver, which at once distinguishes pernicious anæmia post mortem from all other varieties of anæmia. The destruction of the blood differs both in its nature and seat from that found in malaria, and in various forms of hæmoglobinuria. The view can no longer be held that the occurrence of hæmoglobin in the urine simply depends on the quantity of hæmoglobin set free; on the contrary, the *seat* of the destruction and the *form* assumed by the hæmoglobin when liberated are important conditions regulating the presence or absence of that substance in the urine, in any case in which an excessive disintegration of corpuscles has occurred. In paroxysmal hæmoglobinuria, such disintegration occurs in the general circulation, and is due to a rapid dissolution of the red corpuscles. In pernicious anæmia, however, the seat of the destruction is chiefly the portal circulation, more especially that portion of it contained within the spleen and the liver, and the destruction is effected by the action of certain poisonous agents of a cadaveric nature absorbed from the intestinal tract. W. D. H.

Physiological Action of Iron. By SKVORTZOFF (*Brit. Med. Jour.*, **2**, 1888, 727, from *Vratsch*, 1888, 561).—This is a preliminary note giving the results of experiments on dogs, carried out with the view of determining the action of iron on nitrogenous metabolism in a healthy organism. The following are the conclusions:—(1.) Iron has no marked influence on the nitrogenous metamorphosis in a healthy system. (2.) On the internal administration of iron in daily doses, over 0·02 to 0·03 gram, the assimilation of the nitrogenous ingredients of the food decreases, although but slightly (from 98·4 per cent. before the experiment to 97 per cent. during it). (3.) After venesection, the assimilation somewhat increases, both on the administration of iron and without it. (4.) On the administration of iron with food after venesection, the restoration of hæmoglobin proceeds more rapidly than without iron. (5.) The same holds true in regard to the body's weight. W. D. H.

Physiological Action of Ulexine. J. R. BRADFORD (*J. Physiol.*, **8**, 79—85).—Ulexine is an alkaloïd originally prepared by Gerrard from the seeds of the common gorse (*Ulex Europæus*). The hydrobromide was used, since this salt crystallises more readily than any other, and so may be obtained in a greater degree of purity. It was found to have a powerful and wide-spread action, being a nerve and muscle poison, a respiratory poison raising arterial tension, and producing diuresis. The paralysis of respiration is produced by the smallest doses, and is apparently the most important action of the drug. W. D. H.

Albumose, Peptone, and Neurine as Pyrexial Agents. By I. OTT and C. COLLMAR (*J. Physiol.*, **8**, 218—228).—The various albumoses as prepared by Kühne and Chittenden, peptone, papain (probably from the fact that it consists largely of an albumose), neurine, and commercial trypsin (which also contains products of

digestion), all cause in cats and rabbits a rise of temperature or fever. Calorimetric observations show also an increase in heat production. The fever reaches its height about two hours after the injection of the substance into the jugular vein. This, however, does not occur in curarised animals. Pflüger has shown that curare paralyses the thermic nerve-fibres, and so the action of these substances is probably primarily on the nervous system. W. D. H.

Toxic Effects of Albuminous Urine. By J. Teissier and G. Roque (*Compt. rend.*, **107**, 272—275).—In some cases, an increase in the toxic effect of urine affords valuable evidence of increased gravity in the condition of the patient, but this does not always hold good, especially in cases of nephritis. Albuminous urine secreted during sleep is more poisonous than that secreted when awake, whilst with normal urine the reverse is true. In some cases, the toxic effect is in proportion to the quantity of albumin present, but in other cases there is no relation of this kind. No definite connection could be traced between the general composition of the urine and its toxic action. C. H. B.

Toxic Action of Ouabaïn and Strophantin. By E. Gley (*Compt. rend.*, **107**, 348—351).—The characteristic effect of both substances is the rapid effect on the heart of a frog which is arrested in systole. 0·025 of a milligram of ouabaïn produces this effect in six minutes, whilst the same quantity of strophantin requires 12 minutes. With 0·012 milligram of ouabaïn, the arrest takes place in nine minutes. To the rabbit, ouabaïn is twice as poisonous as strophantin, to a dog three times, to a guinea-pig four times. Moreover, strophantin is always less rapid in its action. Both compounds act less energetically when introduced into the stomach than when injected into the veins. C. H. B.

Preventive Inoculation of Rattlesnake Venom. By H. Sewall (*J. Physiol.*, **8**, 203—210).—Repeated inoculation of pigeons with sub-lethal doses of rattlesnake venom (which has been shown by previous observers to be proteïd in nature, see Abstr., 1886, 1057) produces a continually increasing resistance towards the injurious effects of the poison without any apparent influence on the general health of the animals. The efficiency of resistance against the venom gradually fails in the absence of fresh inoculation. In some cases, however, the prophylactic effect of the repeated inoculations was persistent for a period of five months. W. D. H.

Chemistry of Vegetable Physiology and Agriculture.

A Ferment from Putrefactive Bacteria which Dissolves Fibrin. By E. Salkowski (*Zeit. Biol.*, **25**, 92—101).—Fibrin, which had been well washed and exposed for a few days to a temperature of

7—10°, was then kept for many months under chloroform-water (5 c.c. of chloroform to a litre of water), and examined at intervals of every few months. By this means, putrefaction was entirely prevented. The fibrin, however, entered slowly into solution; the proteïds in solution were at first globulin and albumin; later on, these diminished, and albumoses and ultimately peptones appeared.

The different albumoses of Kühne and Chittenden were separated, and full details relating to heat coagulation, precipitation by neutral salts, &c., are given of the various proteïds discovered from time to time. By the end of seven months, at the ordinary atmospheric temperature, there was but little undissolved residue, and that was of a glutinous nature.

The question arises, what is the source of the ferment which brings about this action? It is certainly an unorganised ferment, as living bacteria were excluded throughout the experiment. The ferment must either have been derived from the blood from which the fibrin was obtained, or from the bacteria which contaminated the fibrin after the process of washing.

In order to settle the question, fibrin was first well washed, part was heated in a current of steam, and then put into chloroform-water, the rest was put into chloroform-water directly. In neither case was there any solution of the fibrin, mere traces of proteïd being found in both liquids. If a ferment had been present in the blood, the fibrin in the second case would have entered into solution.

It therefore follows that the ferment had been produced by putrefactive bacteria in the first-described experiment. Such a ferment was, in fact, discovered in the undissolved residue; it was active in an alkaline solution, and was therefore of a nature of trypsin.

W. D. H.

Antiseptic Properties of Mercuric Cyanide, Oxycyanide, and Chloride. By CHIBRET (*Compt. rend.*, **107**, 119—120).—Solutions of mercuric oxycyanide, $HgO,Hg(CN)_2$, are feebly alkaline and give only a slight precipitate with solutions of albumin. They are much less irritating to living tissues than mercuric chloride, and a solution of 1 in 1500 has little action on ordinary metals. It is not decomposed by light. The oxycyanide was found to be six times as effective as the chloride in preserving cultivation liquids. A 1 per cent. solution of the oxycyanide, cyanide, or chloride kills *Micrococcus aureus* at once; a solution 1 : 1000 kills this organism in less than an hour, but a solution 1 : 3000 requires several hours. If agar-agar is immersed for three minutes in 1 : 3000 solution of any one of these salts, it will no longer grow *M. aureus*, and the effect persists for several days.

A solution of mercuric oxycyanide 1 : 1500 is much superior as an antiseptic to a solution of mercuric chloride. C. H. B.

Tobacco and Bacteria. By V. TASSINARI (*Lancet*, **2**, 1888, 729, from *Centr. Bakteriol.*, **4**, 15).—The germicidal virtues of tobacco-smoke were demonstrated by the use of the following apparatus:—In a chamber formed of two glass funnels placed horizontally and connected at their mouths by paraffin, is suspended from a loop of

platinum a small piece of linen, the lower end of which dips in a culture fluid containing the microbes. The chamber is connected at one end by a tube with a cigar or cigarette, and at the other by a tube containing a cotton-wool plug (to act as a filter) with the mouth of the experimenter. The smoke thus thoroughly surrounds the linen, and after the experiment, which lasts half an hour and involves the consumption of 3·5 to 4 grams of tobacco, the chamber is opened and the linen allowed to fall into a test-tube containing fluid gelatin. Seven varieties of pathogenic micro-organisms were thus examined, including those of cholera, anthrax, and pneumonia. In every instance there was very marked delay in the development of colonies in the gelatin compared with that of organisms dealt with similarly but without exposure to tobacco-smoke; and the development of some was entirely prevented. What substance or substances in the smoke have this action is to be the subject of renewed inquiry.

W. D. H.

Nitrification of Ammonium Salts. By H. Landolt (*Bied. Centr.*, 1888, 577—578).—In reply to Frank's criticisms of Plath's work (this vol., p. 521), the author, in conjunction with Plath, repeated the experiments of the latter and found:—(1) That after passing air through concentrated sulphuric acid and through aqueous soda, it gave no nitric or nitrous acid to pure aqueous soda solution even in 14 days; (2) that air thus purified when conducted over calcium carbonate for 14 days gave no trace of oxides of nitrogen; (3) that when the sterilised calcium carbonate was in contact with ammonium sulphate solution the result was the same. Two samples of soil were employed for the next experiments. After washing out with water the nitrates naturally present in the soils, they found that when left in contact with ammonium sulphate solution, nitrites and nitrates were formed when the soils had not been sterilised and were absent when sterilisation had been performed. In another experiment, they found that the natural amount of nitrates in the soils was not increased when they were left in contact with ammonium sulphate solution, having been previously sterilised by ignition and treated with water containing carbonic anhydride, and with phosphoric acid free from nitric acid, in order to neutralise the lime produced by the ignition. They, therefore, conclude that the inorganic constituents of the soil can effect no nitrification of ammonia.

H. H. R.

Growth of Maize and Peas in Nutritive Solutions. By E. Heiden (*Bied. Centr.*, 1888, 622—624).—At the Agricultural Exhibition at Bautzen, a set of experiments was arranged to illustrate the importance of the different plant foods. Maize and pea plants, in sets of five, were grown in nutritive solutions from which one or other of the foods were omitted; a set of each species was also grown in a solution containing all the plant foods. The average heights attained, expressed in centimetres, were as follows:—Lime absent, maize 18·9, peas 21; magnesia absent, maize 30, peas 44; nitrogen absent, maize 32, peas 51; phosphates absent, maize 24, peas 63; potash absent, maize 28, peas 45; all foods present, maize 100, peas 97. Both

species suffered most and died soonest when lime was absent. When iron was absent, the plants were pale, but grew for some time. The author could not get lupins to grow in nutritive solutions.

H. H. R.

Solid Hydrocarbons in Plants. By H. C. S. Abbot and H. Trimble (*Ber.*, **21**, 2598—2599).—When *Cascara amarga* is extracted with light petroleum and the extracted substance crystallised from absolute alcohol, three distinct compounds are obtained. The one crystallises in silky needles, sometimes 2—4 cm. long, melts at 196·2—196·4°, and decomposes at a higher temperature; it dissolves in the usual solvents. When further purified, it contained 87·90 per cent. of carbon and 11·90 per cent. of hydrogen.

15 kilos. of *Phlox carolina* were extracted with light petroleum, and a hydrocarbon of the formula $(C_{11}H_{15})_x$ isolated. N. H. M.

Galactose from Plum-Gum. By R. W. Bauer (*Landw. Versuchs-Stat.*, 1888, 214—215).—The gum (50 grams) was boiled for four hours with 200 c.c. of 55 per cent. sulphuric acid, filtered, neutralised with calcium carbonate, evaporated and extracted with absolute alcohol. In the residue, arabinose, dextrose, and galactose were found, the latter to the amount of 0·844 gram, having a rotatory power of $[\alpha]_D = 78·07$ at 12° in a tube 200 mm. long. E. W. P.

Explanation of the Changes which the Nitrogenous Matters in Silage undergo. By E. Schulze (*Landw. Versuchs-Stat.*, 1888, 195—208).—After reviewing the researches of others in the same direction, the author draws this conclusion from their results, that in young plants which have been cut and kept moist in the dark, the albuminoïds are converted into amides. He then details his own experiment with timothy grass and oats, when albuminoïds decreased, but amides increased; in particular, asparagine was formed in very large quantity, although it was not present previous to siling. It appears that the extent of conversion is dependent on the character of the plants employed, and on the stage of growth at which the plants are cut, and that the asparagine, together with the other amides, may by the continued action of the fermenting organisms be further converted into ammonium salts and amido-acids (leucine, tyrosine, &c.). When then the silage is dried, dissociation of the ammoniacal salts occurs and ammonia is lost, and this accounts for the deficit of nitrogen which many have observed. If the plants have been previously dried before fermentation, no asparagine is formed at the expense of albuminoïds. at any rate the proteïds are but very slowly attacked; on the other hand, non-nitrogenous compounds are more readily attacked, and also the amides, consequently such silage must and does contain ammonia, and asparagine, &c., will be absent: these statements are all supported by analytical data. The loss of nitrogenous nutritive matter is therefore due to the formation of asparagine, &c., at the cost of albuminoids, and to the retardation of the action of the fermenting organisms, but which of the two causes has most influence is dependent on the character of the plant, and its period of growth and development when cut. The changes which

occur when sweet ensilage is made under pressure at 60° have still to be investigated. E. W. P.

Absorption of Nitrogen by Leguminosæ. By E. Bréal (*Compt. rend.*, **107**, 397—399).—In several species of leguminosæ, the total nitrogen in the plant is about twice as great as that originally present in the seeds. If, however, the roots are covered with tubercular nodosities the amount of nitrogen is much greater. In one case with lucern it was 25 times as great as in the seed. These tubercles can be transferred from one plant to another by inoculation. They contain a whitish liquid full of microscopic, highly refractive, rounded granules, and moving filaments which resemble bacteria.

C. H. B.

Relation between Atmospheric Nitrogen and Vegetable Soils. By T. Schloesing (*Compt. rend.*, **107**, 290—296).—In previous experiments (this vol., pp. 747 and 870), the oxygen and nitrogen were in contact with the soil in closed vessels without any communication with the outside air. Two new series of experiments were made, in one of which a slow current of air was passed continuously into the vessel containing the soils, whilst in the other the soils were freely exposed to the air. Seven varieties of soil were used, namely: (1) A very fertile alluvial mud from Boulogne-sur-Seine manured with dung and night soil; (2) its sub-soil from a depth of 0·6—0·7 m.; (3) a non-calcareous argillaceous sand from Neauphle; (4) its sub-soil at a depth of 0·4 to 0·5 m. (5, 6, 7), the soils from Grenelle, Fouilleuse, and Montretout respectively, used in the former experiments. All the soils were passed through a sieve and allowed to dry spontaneously before being analysed. The amount of moisture left in the soils varied from 13 to 18 per cent. The experiments extended from February, 1886, to April, and in some cases July, 1888. Nitrification went on as usual, and there was a decrease in the amount of ammonia. A small quantity of the carbon was converted into carbonic anhydride.

There was no appreciable difference between the total amount of nitrogen present in the soil at the end of the experiment and that originally existing in it. The differences were sometimes positive and sometimes negative, the maximum in each case being +0·53 and −0·57 miligram of nitrogen per 100 grams of soil. This result confirms the conclusion drawn from the previous series of experiments.

C. H. B.

Absorption of Nitrogen by Vegetable Soils and by Plants. By Berthelot (*Compt. rend.*, **107**, 372—378).—In recent experiments which have given negative results, the soil has been treated merely as a chemical compound, without due regard to the bacteria which are present.

Three slightly calcareous argillaceous soils were used in the experiments described in this paper, two being somewhat rich in nitrogen, whilst the other contained only a very low proportion. In each soil, six species of leguminosæ (vetch, lupin, clover, lucern, &c.) were sown, and the experiments were made under four different conditions,

namely, with free exposure to air; under cover with free circulation of air; in closed vessels into which was passed each day 50 litres of filtered air free from ammonia and 1 litre of carbonic anhydride; in vessels hermetically sealed. Similar experiments were made with the same soils without living plants. The soil in each case was inoculated with bacteria which are supposed to be active in promoting the absorption of nitrogen. The experiments described relate to the series without plants and those with lupin and vetch, the latter being gathered when they reached maturity.

In all cases there was a distinct gain of nitrogen, the minimum being 0·083 gram or 2·1 per cent., and the maximum 0·9865 or 27·2 per cent. The soil growing no plants shows as well-marked an increase as soil with plants. With lupins, and with vetches in closed vessels, the gain of nitrogen takes place solely in the soil itself, the nitrogen in the plant being no greater than that in the seed, but in the case of vetches growing with free exposure to air, there is a very large increase of nitrogen and a considerable proportion of this is in the plant.

All these experiments were made with soils not very rich in nitrogen. With soils containing a large proportion of nitrogen, the increase is by no means so well marked, and in some cases there is even a decrease. With clover, any gain in nitrogen is almost entirely confined to the plant. C. H. B.

Composition of Barleys grown in Württemberg in 1887. By Behrend (*Bied. Centr.*, 1888, 620—622).—Saale barley and v. Trotha's Chevalier barley were grown and compared with local kinds of barley. Although the composition of the 89 samples examined varied very much, the starch ranging from 69·1 to 59·8 per cent., and the albuminoïds from 8·1 to 11·7 per cent. (nitrogen from 1·30 to 1·87 per cent.), yet the average composition of the barleys of one kind did not differ from the averages given by the other kinds; hence the inference is drawn that the influence of soil, cultivation, &c., on the composition is so great that the influence due to the kind of seed may be neglected.

A classification of the barleys according to quality was made by experts, and on examining the composition of the samples in these classes, it appeared that the high class barleys were in the average the richest in starch and the poorest in albuminoïds, and the low class barleys were just the reverse.

On placing the samples in groups containing about the same amount of starch, it was seen that the albuminoïd averages for the groups were much about the same (10·0 to 10·3 per cent.) for those groups where the starch averages were not over 64·4 per cent., but for the groups with more starch than this, the albuminoïd averages fell continuously as the starch averages rose. H. H. R.

Egyptian Cigarettes. (*Lancet*, **2**, 1888, 785—786.)—This is the report of an analytical commission appointed to inquire into the alleged poisonous nature of Egyptian cigarettes. Five varieties were examined, and the results of analyses, microscopical and chemical, are

as follows:—The cigarettes are made of genuine tobacco and are free from opium. The ash contains a trace of copper which is probably due to the metallic lettering of the paper. Arsenic is absent.

Some proportion of Smyrna or Saloûk tobacco is commonly incorporated with the Turkish, and this has an injurious action on the throats of some people, if it is present in excess. W. D. H.

The Composition of Stable Manure. By F. Sestini (*Gazzetta*, **18**, 44—54).—This paper contains the results of the analysis of a large number of samples of stable manure from the ox and from the horse, various kinds of litter being used. From the composition of these manures, the author considers that they are wanting in phosphoric acid rather than in potassium salts, and recommends the use in addition of artificial phosphatic manures. The nitrogen exceeds the 5 per cent. usually regarded as the theoretical amount necessary for a good manure, but it is not in a form in which it can be easily assimilated. C. E. G.

Lime and Ash in Tyrolese and other Wines. By E. Mach (*Bied. Centr.*, 1888, 638—640).—Determinations of lime and ash in a large number of wines.

Analytical Chemistry.

Instruments for Measuring Liquids. By Greiner and Friedrichs (*Zeit. anal. Chem.*, **27**, 470).—The authors have adapted the glass three-way cock with oblique perforations to a burette, which thus in one position of the plug is filled from below, and in the opposite position delivers its contents through a jet. For measuring out successive equal quantities of a liquid, the three-way cock is attached to a pipette made to contain the requisite volume when filled to overflowing. The liquid which overflows is caught in a funnel surrounding the stem of the pipette, and runs away through a side tube. M. J. S.

Apparatus for Quantitative Analysis. By G. Neumann (*J. pr. Chem.* [2], **38**, 85—91).—The author has improved his "hydrometer" (Abstr., 1887, 1140) by having a bulb of 100 c.c. capacity blown on the burette underneath the stop-cocked funnel. The stop-cock of the latter is three-way, so that connection may be made with a decomposing flask. A similar burette without bulb and stop-cocks is ground into the funnel, and quantities of gas smaller than 100 c.c. may be measured in this. The apparatus may be used as a Bunte's burette, as a Zulkowsky and Schiff's nitrogen measuring tube, and as a Lunge's nitrometer.

A funnel is described, having an air-tight cover with a cork and bent tube running through it, for the rapid washing of precipitates.

A dropping funnel with a three-way stop-cock, through which a gas as well as the liquid may be passed, and a peculiar litre flask, are also described. All are figured in the original paper. A. G. B.

Metallic Felt Filters. By C. E. MUNROE (*Chem. News*, **58**, 101—102).—Ammonium platinochloride is placed while moist into a Gooch platinum crucible; the crucible is cleaned, dried, and carefully ignited; the resulting layer of platinum sponge forms a good filtering surface. If cracked, it may be repaired by applying some of the fresh salt to the crack and re-igniting. D. A. L.

Determination of Sulphur in Coke. By L. BLUM (*Zeit. anal. Chem.*, **27**, 445—452).—Oxidation by wet reagents (hydrochloric acid and potassium chlorate, hydrochloric acid and bromine, aqua regia), although furnishing results which agree amongst themselves, in no case gives the whole of the sulphur present, but only a small proportion. The total sulphur is best determined by fusion; 1 part of the coke is fused with 4 parts of sodium carbonate, 8 parts of potassium nitrate, and 16 parts of sodium chloride. The mass is dissolved in hydrochloric acid, evaporated to dryness, redissolved in hydrochloric acid, filtered from silica, and the sulphuric acid then precipitated by barium chloride. In 10 samples of Belgian and Westphalian coke, the total sulphur ranged from 0·907 to 1·147 per cent. The sulphur found by wet oxidation is that present as metallic sulphides. The remainder, which exists as an organic compound, is, in the author's opinion, the more injurious of the two in metallurgical operations. M. J. S.

New Methods for Estimating Sulphur in Steel and in Iron for Steel-making. By J. O. ARNOLD and H. J. HARDY (*Chem. News*, **58**, 41—44).—0·2 gram of the steel, a few pieces of pure stick zinc, and about 20 c.c. of water are placed in a flask of about 200 c.c. capacity, connected by means of two tubes and india-rubber joints with a graduated cylinder containing 1 : 4 sulphuric acid on one side, and with a tube marked at 30, 40, and 50 c.c. containing 30 c.c. of a solution of 25 grams of sodium hydroxide in a litre of water. The tube to the acid terminates at both ends with a fine aperture, whilst the end of the tube in the soda is also contracted. The water in the flask is boiled to expel air, the soda tube is then closed by clipping the india-rubber tube, and by removing the lamp 15 c.c. of acid is allowed to pass into the flask, when the india-rubber tube on the acid tube is also closed by clip. As soon as the pressure of hydrogen is sufficient, the soda tube clip is removed, and the gas is allowed to bubble through the soda, which absorbs all the hydrogen sulphide. The flask is heated until the steel is dissolved, when the acid continues to act on the zinc, the hydrogen driving the last portions of the hydrogen sulphide into the soda. The action is continued until the cooled contents in the soda tube measure 40 c.c., then 10 c.c. of a dilute acid solution of lead acetate is added, and the colour compared with a standard prepared at the time in a similar tube from pure lead acetate and hydrogen sulphide, from

which the amount of sulphur in the steel is calculated. The operation lasts less than half an hour, and with steels containing 0·01—0·04 of sulphur the results are within 0·01 per cent.; with 0·06—0·1 steels they are within 0·02 per cent., and with 0·11 to 0·14 steels within 0·03 per cent. Hence when good approximate results are required, the method answers well.

By another and somewhat more elaborate method results are obtained which are constant and accurate within less than 0·01 per cent. A flask supported on a sand-bath is connected with a reservoir of pure hydrogen and a series of 15 small tubes. The first acts simply as a condenser; the others contain each sufficient lead acetate solution (2 c.c. of a solution of 1·812 gram of crystallised normal lead acetate in 1000 c.c. of water acidified with acetic acid) to form lead sulphide with the hydrogen sulphide produced by 0·01 per cent. of sulphur in 2 grams of steel. This quantity of the metal is weighed into the flask, 30 c.c. of water is added, and the tube from the hydrogen reservoir just dips under it; 1 litre of hydrogen is aspirated through the apparatus to expel air, 30 c.c. of 1 : 4 sulphuric acid is added through a tap funnel, the whole carefully heated, and when the action is finished another litre of hydrogen is aspirated through, and the number of bottles discoloured is counted, and as no hydrogen sulphide passes out of a tube until all the lead is converted into sulphide, the number of discoloured tubes represents hundredths per cent. of sulphur in the steel. If the last tube is dark ruddy-brown, it may be considered as half the quantity, whilst a yellow-brown coloration indicates a trace of hydrogen sulphide. Neither copper nor phosphorus if present interfere with this process. The current of hydrogen drives all the hydrogen sulphide out of the first tube of the 15. D. A. L.

Estimation of Sulphur in Iron and Steel. By J. J. Morgan and others (*Chem. News*, **58**, 63, 70, and 95).—Morgan draws attention to weak points in various methods, but regards methods based on the oxidation to sulphuric acid by means of nitric acid as the most trustworthy; he nevertheless places great confidence in Parry's method (Abstr., 1887, 1140). J. O. Arnold and H. J. Hardy do not, however, find the latter method capable of great accuracy, inasmuch as precipitation of lead sulphide occurs, which they attribute to the disturbance caused by the bubbling of the hydrogen sulphide into the liquid. B. W. Winder supports the views of the latter authors, commends their self-registering method (preceding Abstract), and refutes some of Morgan's experiments. D. A. L.

Sources of Error in Determinations of Nitrogen by Soda-lime, and Means for avoiding them. By W. O. Atwater (*Amer. Chem. J.*, **10**, 262—282; compare this vol., p. 990).—After reviewing the work of numerous analysts and describing his own experiments, the author concludes that there is no loss of ammonia either by dissociation or oxidation (1) if the tube is closely packed with soda-lime for a length of 12 cm., and this be heated before the distillation proper begins; (2) if the time of combustion is not more than three quarters of an hour; (3) if the tube is heated only to dull redness

and is allowed to cool somewhat before the last traces of ammonia are drawn out by a current of air. The insufficient mixing of the substance with soda-lime, the leaving of open channels above the mixture, protracted heating and employment of high temperatures, all tend to produce too low results. Kjeldahl's method is decidedly preferable for convenience, &c.; it is best, however, to check the two methods against one another, and with some substances it is necessary to control both by the absolute method. H. B.

Estimation of Nitrogen in Vegetable Soils. By BERTHELOT and G. ANDRÉ (*Compt. rend.*, **107**, 207—209).—The estimation of nitrogen in soils presents some difficulty when part of the element is in the form of nitrates, and the various methods which have been devised for the estimation of the total nitrogen under these conditions are either difficult of execution or are liable to considerable errors.

The difficulty may be avoided in the case of true vegetable soils by treating them with four times their weight of cold water and thus removing the nitrates. This treatment removes at most a few milligrams of organic nitrogen per kilo., a quantity much smaller than the errors of the determination. C. H. B.

Estimation of Carbon and Nitrogen in Vegetable Soils. By T. SCHLOESING (*Compt. rend.*, **107**, 296—301).—The ordinary method of combustion by means of oxygen and cupric oxide gives satisfactory results in the determination of the carbon in soils, but in order to avoid errors arising from the presence or formation of alkaline carbonates, the carbonates existing in the original soil and in the residue from the combustion should be carefully determined.

Nitrogen is most accurately estimated by Dumas' method, but the greatest care is required in sampling. In order to reduce the errors of experiment, the author operates on comparatively large quantities of soil. The combustion tube is 2 metres in length, and is drawn out at the end connected with the pump. This end of the tube is connected with a small distilling flask, in which the water is condensed, and the side tube of the flask is connected with the pump, the flask and joints being immersed in a beaker containing water. Commencing from the drawn-out end, the tube is filled with a layer of granulated cupric oxide 10 cm. in length, a layer of granular reduced copper 20 cm. in length, a second layer of cupric oxide 28 cm. long, and a tube, 2 cm. long, containing about 10 grams of pure lead carbonate, the different substances being separated by plugs of asbestos. The remainder of the tube, except about 15 cm. at the other end, is filled with the soil. The other end of the tube is connected with a retort containing pure potassium chlorate, the joint being surrounded by mercury.

The tube is made vacuous, the pump is stopped, and the potassium chlorate is heated until the gas in the tube is again at atmospheric pressure. A vacuum is again made, and the chlorate is heated gently in order to produce a slow current of oxygen. The lead carbonate is now heated until the gas issuing from the tube is completely absorbed by potash. The pump is then stopped, and the heating of the carbo-

nate continued until the tube is filled with carbonic anhydride at the ordinary pressure. The front part of the tube is now heated, and when the copper and copper oxide are red-hot, the soil is gradually heated. Destructive distillation of the organic matter takes place, and as soon as the soil is heated to redness, the potassium chlorate (which has been kept fused) is heated very carefully in order to obtain a regular stream of oxygen. As soon as combustion is complete, the tube is again made vacuous. The gas thus obtained is almost pure nitrogen, but sometimes contains a combustible gas amounting to 0·2 per cent., and occasionally even to 2 per cent. The presence of any nitric oxide is quite exceptional, and indeed was only observed in one instance. In the first stage of the combustion, the tube is filled with a reducing atmosphere, and it would seem that the gaseous oxygen is unable to oxidise the nitrogen during the subsequent combustion of the carbonised matter. C. H. B.

The Azotometric Method of Soil Analysis. By A. Baumann (*Zeit. anal. Chem.*, **27**, 457—461).—A rejoinder to Knop (this vol., 533). The author still maintains the uselessness of the azotometric method of determining the ammonia in soils, and recalls the experiments by which he showed that the contraction observed is due to humus, and is not prevented by the addition of even large quantities of borax solution. M. J. S.

Estimation of Nitric Acid. By H. Wilfarth (*Zeit. anal. Chem.*, **27**, 411—433).—In this modification of Schlösing's method the nitric oxide is absorbed by a mixture of sodium carbonate and hydrogen peroxide in presence of air. The apparatus consists of a flask in which the nitrate is boiled with ferrous chloride and hydrochloric acid. This is followed by a miniature Woulff's bottle for the condensation of the steam and the greater part of the hydrochloric acid. The gas next passes, through a Bunsen's caoutchouc valve, into a test-tube fitted as a washing bottle, and containing sodium carbonate, and thence to the absorption apparatus, which consists of a flask and a set of bulbs of peculiar form. The air is expelled from the decomposition flask by carbonic anhydride, which by means of a three-way cock can be diverted to the Woulff's bottle, and employed to drive back the condensed liquid into the decomposition flask. A branch tube serves for introducing the substance and reagents, and for removing the contents of the flask after the analysis. Not more than 0·5 gram of nitrate should be used. This is dissolved, and drawn into the flask, together with 20 c.c. of ferrous chloride (saturated solution), and 60 c.c. of hydrochloric acid (1·124), after filling the whole apparatus with carbonic anhydride. The absorption flask and bulbs are charged with a mixture of 20 c.c. of sodium carbonate solution (26 grams per litre), and 50 c.c. of hydrogen peroxide. The contents of the decomposition flask are boiled for five minutes, and then carbonic anhydride is passed for 10 to 15 minutes, a filter moistened with a solution of metadiamidobenzene being placed over the outlet of the bulb apparatus to detect any escape of nitrogen oxides. The soda-washing bottle is now disconnected from the absorption flask, and

the filter removed to that position to ascertain whether the last traces of nitric oxide have been swept out. When this is found to be the case, the absorption apparatus is set aside for an hour to complete the conversion of the nitrogen oxides into nitric acid. Its contents are then united; a measured excess of standard sulphuric acid is added; the mixture is boiled for 10 minutes, cooled, and the free acid (consisting of the nitric acid formed and the excess of sulphuric acid added) is titrated with soda (not baryta) solution, using an indicator (cochineal, litmus, lacmoid), not affected by the excess of hydrogen peroxide. Very satisfactory results have been obtained when using as little as 0·04 gram of potassium nitrate. Each determination occupies 25 to 30 minutes. M. J. S.

Phenol and some Allied Substances as Tests with Concentrated Sulphuric Acid for Nitrites, Nitrates, and Chlorates in Aqueous Solution. By D. Lindo (*Chem. News*, **58**, 1—3, 15—17, 28—29).—The author employed the following solutions in his numerous experiments. Phenol:—10 c.c. of Calvert's No. 1 fused, dissolved in some water mixed with 25 c.c. of 97 per cent. alcohol, and made up to 100 c.c. with water. Orcinol:—5 grams dissolved in water, and made up to 100 c.c. Sulphuric acid, pure commercial, sp. gr. 1·837 at 26°. Nitric solution:—1·8723 grams of potassium nitrate made up to 100 c.c. = solution 100 N_2O_5. Nitrous solution:—0·4053 gram of silver nitrite dissolved in 100 c.c. of boiling water, mixed with 0·160 gram of sodium chloride in solution, allowed to cool, made up to 200 c.c., agitated, and when settled and filtered = solution 2000 N_2O_3. Chloric solution:—1·624 grams of potassium chlorate dissolved and made up to 100 c.c. = solution 100 Cl_2O_5. The solutions of hydrochloric acid used were *a*, containing 26·16 per cent., *b*, 13·08 per cent., and *c*, 4·88 per cent. The sulphurous acid solution contained about 4 per cent. of the gas. The ferric chloride about 36·5 per cent. of the salt, the ferrous sulphate 10 per cent., and the copper sulphate 0·5 per cent. of the metal. In all experiments, 0·5 c.c. of the solution to be tested was mixed with one drop of phenol or two drops of orcinol solution, except with chlorates, when 1 drop of orcinol was employed; 2 c.c. of sulphuric acid is then run down the side of the tube so as to obtain bands of colour at the contact of the liquids. When sulphurous acid is used it is always added first, the phenol or orcinol being always the last added before the sulphuric acid. The following are some of the results obtained.

Phenol and nitrites:—A 200,000 N_2O_3 solution yields a faint pink band at first, faint green below; pink band very distinct in half an hour, but after five hours the colour has faded away. 50,000 N_2O_3 solution gives good red and green bands at once, fading after five hours. 2000 N_2O_3 solution very intense red and green bands in 24 hours, the latter changes to blue and the red to dingy purple. Solutions of intermediate strengths give intermediate reactions, and even 400,000 N_2O_3 gives a very faint band. Hydrochloric acid impedes or retards the reaction, so that 1 drop of solution *a* renders the colour with 200,000 N_2O_3 indistinct, and 2 drops upset the reaction with 50,000 N_2O_3 solution, and make the 10,000 give a light red band with

yellowish-green below. Two drops of sulphurous acid, on the other hand, prevent the colour forming even with the 2000 N_2O_3 solution; but with the addition of copper sulphate (see below) a slight pink band is obtained after two or three hours, presumably due to a trace of N_2O_5.

Orcinol and nitrites:—One of nitrite in 1,000,000 of water gives a faint but uncertain indication after some time; 500,000 N_2O_3 gives a very faint pink after four hours, becoming more distinct in nine hours; 10,000 N_2O_3 solution gives a strong orange band at once, becoming almost opaque after some time. The colour is permanent. Hydrochloric and sulphurous acids both interfere with the reaction, but copper sulphate with hydrochloric acid increases the delicacy; 1,000,000 N_2O_3 + 1 drop of copper sulphate + 2 drops of orcinol + hydrochloric acid, sharp orange-pink band in 10 minutes.

Phenol and nitrates:—No reaction with 1 N_2O_5 in 20,000 of water. 10,000 N_2O_5 faint, pink band becoming broader and less definite, ultimately fading; 1000 N_2O_5, intense red band, pale green below becoming very dense. Intermediate and stronger reactions are given in the original. Phenol alone appears to be but a poor test for nitrates, but in the presence of hydrochloric acid it becomes very delicate; by adding one drop of solution *c* 100,000 N_2O_5 gives a faint pink at once, which becomes distinct in 20 minutes, spreads and ultimately fades; 10,000 N_2O_5, a sharp, crimson band at once, spreading without losing intensity for some hours, and faint green below, soon fading. 1000 N_2O_5, very dense red, green below. Sulphurous acid does not interfere with this test, or with the corresponding one with orcinol. Neither ferric chloride nor copper sulphate disturbs this reaction, in fact, the latter rather improves it with highly dilute N_2O_5 solutions; but ferrous sulphate is fatal, so that with 1 drop of the solution, and hydrochloric acid 10,000 N_2O_5 gives no reaction, and 5000 is very indistinct.

Orcinol and nitrates:—The remarks which apply to phenol are mostly true of orcinol, but with the addition of hydrochloric acid the latter is more sensitive, indicating by a faint pink colour 1 N_2O_5 in 200,000 in three hours, and with extreme faintness 1 in 500,000. The colour is permanent, and orcinol may be regarded as very superior to phenol for detecting traces of nitrate in the presence of large quantities of hydrochloric acid or chlorides. Ferric chloride is objectionable, but neither sulphurous acid nor ferrous sulphate interferes with the reaction, whilst copper sulphate makes it more delicate; 500,000 N_2O_5 with hydrochloric acid, 1 drop of copper solution and 2 drops of orcinol gives sharp bands in five minutes.

With chlorates, phenol gives a pale yellow band with blue below, the latter soon fading with 10,000 Cl_2O_5; with 4000, the blue soon changes to dingy green, with 2000 the blue is dingy and becomes dingy green; with 1000 and upwards no blue is developed. Orcinol is somewhat more delicate, 100,000 Cl_2O_5 giving a faint transient blue band, which becomes more distinct as the strength of the solution increases, so that with 10,000 Cl_2O_5 a beautiful blue band is produced, soon acquiring the colours of the rainbow, 4000 gives a similar effect, but not so good, owing to interference of a deep green coloration: 1000 gives an intense green, soon turning brown, and 100 only a pale yellow. These changes are not observed with other oxidising agents, and therefore this may possibly

become a characteristic test for chloric acid. Hydrochloric and sulphurous acids interfere slightly with this reaction.

These reactions have been applied to the detection of nitrites, nitrates, and chlorates in the presence of each other, and the author has tested this point by various mixtures made from his solutions. With nitrites and nitrates in various proportions there seems to be no difficulty in detecting them both; the nitrite is first identified, the mixture is then treated with a drop or two of sulphurous acid to destroy the nitrite; to ensure the complete destruction of the nitrite the solution of the mixture should be acid; it is then tested again with hydrochloric acid and orcinol, or if great delicacy is desired, with the addition of copper sulphate, when mere traces of nitrate give a coloration. With nitrites and chlorates, the red coloration with phenol and sulphuric acid due to nitrites is first obtained; and then, after treatment with sulphurous acid, the chlorate may be detected by the blue colour with orcinol and sulphuric acid. The results show that chlorate can be detected when mixed with a considerable excess of nitrite after the latter has been destroyed with sulphurous acid, but nitrites cannot be detected by this method if the chlorate is greatly in excess. With mixtures of chlorates and nitrates, the former cannot be detected if the latter are in considerable excess; but, on the other hand, a small quantity of nitrate can be detected in the presence of a large quantity of chlorate, since by allowing the sulphurous acid to act for about 15 minutes the chlorate is completely destroyed, whilst the nitrate remains and gives its colour reaction in the usual manner; when, however, the chlorate is not in great excess the nitrate may even be detected with hydrochloric acid, phenol, and sulphuric acid, without previously destroying the chlorate. When all three are present in the same solution, they can be tested by these reactions only when the proportions indicated above are not exceeded; for instance, a mixture of equal volumes of 10,000 N_2O_3, 10,000 N_2O_5, and 10,000 Cl_2O_5 would give the red band with phenol and sulphuric acid for nitrites; a blue band with 1 drop of sulphurous acid and 1 drop of orcinol for chlorates; and by treatment with 2 drops of sulphurous acid and 1 drop of hydrochloric acid, allowing to remain 15 minutes, copper sulphate and orcinol would give the orange bands due to nitrate.

The presence of iodides, bromides, and even chlorides in large quantities would prevent the detection of nitrites, nitrates, or chlorates by this method, if these are present, therefore, the solution must be precipitated with silver sulphate. Thymol and α-naphthol do not give distinct and characteristic reactions for these salts under the conditions given in the paper; but the following test is recommended by the author for nitrite in very dilute solutions: two or three drops of the alcoholic α-naphthol and one or two drops of 1 : 1 sulphuric acid added to 10 c.c. of 1 in 1,000,000 nitrite solution, gives in a few minutes a pale, greenish-yellow colour; with much stronger solutions, a precipitate separates, and the colour of the liquid approaches orange.

For the sake of comparison, the following substances were tested with sulphuric acid and 1 drop of phenol or 1 drop of orcinol to 0·5 c.c. of the solution :—Potassium ferricyanide in strong solution gave a brown band with sulphuric acid alone, in weak solutions it gave a brown band

with phenol, and yellow turning brown with orcinol. $\frac{N}{50}$ Potassium permanganate gave with *phenol* a broad, turbid, light reddish-brown band; and with *hydrochloric acid*, in addition, a broad, pale yellow upper band with a narrow, ruddy lower band, changing to bright vermilion; with *orcinol*, it gave reddish upper and dull green lower band, and with *hydrochloric acid* in addition a dull orange upper and emerald-green lower band; in presence of two drops of sulphurous acid, no reaction. Potassium dichromate, 0·5 gram per litre of water, with or without hydrochloric acid, gave with *phenol* a brown band soon becoming lighter, with *orcinol*, a dull, prismatic band; in presence of sulphurous acid no reaction: distinction from chlorates. Hydrogen peroxide, 3 vols. strength diluted with 20 vols. of water, gave with *phenol* a reddish upper, and dull green lower band, and with *hydrochloric acid* a very faint, purple band, changing to pink; with *orcinol*, a sharp reddish band, changing to dull orange above, olive-green below, and with *hydrochloric acid* a purple upper, weak olive-green lower band; no reaction in presence of sulphurous acid.

The above reactions refer only to dilute solutions as indicated in the paper; very different results would be obtained with either stronger reagents or stronger solutions. The average temperature throughout these experiments was about 26°.

The following is a convenient reagent for detecting nitrites or nitrates:—Dissolve 2·5 grams of orcinol in a mixture consisting of 25 c.c. of 2 per cent. copper sulphate solution, 1 c.c. of pure sulphuric acid, and 25 c.c. of pure 15 per cent. hydrochloric acid, and filter; 2 drops of this are added to 0·5 c.c. of the solution to be tested, and 2 c.c. of sulphuric acid run in. If the reaction, as already described above, occurs in presence of sulphurous acid, nitrates are present. (See also next Abstract.) D. A. L.

Griess' Sulphanilic Acid Test for Nitrous Acid modified. By D. Lindo (*Chem. News*, **58**, 40).—The author has made experiments with thymol, orcinol, and α-nahpthol as well as with phenol; he confirms Gambelli's results as regards colour and the advantage of making the solution alkaline (Abstr., 1887, 533). He takes 5 c.c. of nitrite solution, adds 1 drop of a saturated solution of sulphanilic acid in 1 : 5 sulphuric acid, 1 drop of phenol or thymol, and after 10 minutes mixes well with 1 c.c. of ammonia. Thymol gives an orange-yellow not so defined as phenol. The limit of this method, using 5 c.c. of solution, is 1 part of N_2O_3 in 10,000,000 of water. The author suggests the use of the method in the reverse order as a test for phenols. Orcinol gives as good a reaction as phenol, but becomes coloured of itself on exposure to the air in presence of alkali, and is therefore untrustworthy. D. A. L.

Qualitative Test for Phosphoric Acid of Mineral Origin, in presence of Phosphoric Acid of Animal Origin in Artificial Manures. By N. V. Lorenz (*Chem. Centr.*, 1888, 1039).—The test depends on the fact that mineral phosphates all contain much more fluorine than bones. 30 grams of the manure are treated in a beaker with 15 c.c. of concentrated sulphuric acid and covered with a watch-

glass, on the underside of which is a drop of water. The silicon fluoride evolved in the case of a mineral phosphate, causes a deposition of silica in the drop of water. J. W. L.

Volumetric Estimation of Phosphoric Acid by means of Molybdic Acid. By A. GRETE (*Ber.*, **21**, 2762—2765).—Phosphoric acid can be estimated volumetrically as follows:—A known quantity of the solution is neutralised, using methyl-orange as indicator, and any precipitate which may be produced, dissolved by a few drops of nitric acid. An equal volume of a 75 per cent. solution of ammonium nitrate, about 1 gram of sodium sulphate, and 1 c.c. of a solution of gelatin are added, the whole heated at about 80—90°, and titrated with a solution of ammonium molybdate until no further precipitation occurs. The mixture is constantly shaken during titration, and from time to time a little of the gelatin solution is added as long as it produces a precipitate. When the titration is almost completed, the solution is heated and stirred until the precipitate becomes granular; the end of the reaction is then easily observed. If too much molybdate has been run in, a few cubic centimetres of phosphoric acid can be added and the solution again titrated.

The molybdate solution is prepared by mixing 1 kilo. of ammonium molybdate, dissolved in ammoniacal water, with 100 grams of gelatin which has been previously boiled for some time with dilute nitric acid and pouring the slightly alkaline, yellow liquid into about 3 litres of concentrated nitric acid (sp. gr. 1·2). The mixture is diluted to 10 litres, kept for several days with frequent shaking, filtered, and standardised.

The gelatin solution is prepared by boiling 100 grams of gelatin with water and about 50 c.c. of nitric acid, and diluting to 1 litre. A few cubic centimetres of this solution suffice for the precipitation of 0·1 gram P_2O_5.

The analysis can be carried out in 20 to 30 minutes, and the results are very satisfactory. F. S. K.

Determination of Carbon in Iron. By L. L. DE KONINCK (*Zeit. anal. Chem.*, **27**, 463—464).—In the process in which the iron is dissolved by a mixture of cupric and ammonium chlorides (Creath) and the residue oxidised by sulphuric and chromic acids (Ullgren), it is necessary to wash the chlorides out of the residue very completely, otherwise chlorine will be evolved with the carbonic anhydride. The addition of an excess of silver sulphate to the mixture in the oxidation flask obviates this difficulty, not a trace of chlorine being evolved. M. J. S.

Polaristrobometric Analysis of a Mixture of Sodium and Potassium Chlorides. By F. SCHÜTT (*Ber.*, **21**, 2586—2594).—The method of analysis which is based on the influence of inactive compounds on the rotation of polarising substances, forms a ready means of determining the relative amounts of sodium and potassium in solutions from which other substances have already been separated.

The metals are converted into chlorides, and 10 grams of the well-dried mixture put into a Mohr's 50 c.c. flask; 11 grams of potassium

tartrate (with $\frac{1}{2}$ mol. H_2O) previously dried at 110° is then added, the whole dissolved in warm water, cooled to 20°, and the flask filled exactly to the mark. It is well shaken and filtered quickly; the filtrate is then polarised in a 400 mm. tube at 29°. The rotation is first determined with pure sodium chloride and potassium tartrate (22 grams of NaCl, 22 grams of $C_4H_1K_2O_6+\frac{1}{2}H_2O$ dissolved to 100 c.c.), then with pure potassium chloride, and the difference calculated. Tables are given by means of which the percentage of potassium can be found after the rotation has been determined; also a table showing actual percentage amounts of potassium chloride and the amounts as determined (from 5 to 95 per cent.); the greatest error is ± 0·5.

N. H. M.

Estimation of Lithium as Fluoride. By A. Carnot (*Compt. rend.*, **107**, 237—240).—Freshly precipitated lithium fluoride requires for complete solution at the ordinary temperature 800 parts of pure water and 1900 parts of a mixture of equal volumes of water and ammonia solution. In presence of a small quantity of ammonium fluoride, the solubility is still further reduced, probably because the dissociation of the lithium fluoride is prevented, and solution of the precipitate requires 2100 parts of water, 3000 parts of a mixture of 3 vols. water and 1 vol. ammonia solution, and 3500 parts of a mixture of equal volumes of water and ammonia. Sodium fluoride requires 70 parts of the last mixture for complete solution, but the potassium, cæsium, and rubidium compounds are much more soluble.

The ammonium fluoride required is purified by boiling it for a short time with twice its volume of ammonia solution, filtering off the precipitated silica, and preserving the concentrated ammoniacal solution.

The solution containing, say, a few decigrams of a lithium salt with not more than 10 or 15 times its weight of other alkaline salts, is concentrated to a few c.c., mixed with 15 to 20 c.c. of ammonium fluoride and ammonia, and allowed to remain overnight. The precipitate is washed two or three times with a few c.c. of water containing ammonium fluoride and ammonia, dried, heated to expel excess of ammonium fluoride, mixed with excess of sulphuric acid, and the lithium weighed as sulphate. The volume of the filtrate and the washings should be measured. Since part of the liquid is in contact with the precipitate for a short time only, it may be taken that 10 c.c. contains 2 mgrms. of lithium fluoride, approximately equivalent to 4 mgrms. of the sulphate. The examples given are very satisfactory.

C. H. B.

Estimation of Lithium in Mineral Waters. By A. Carnot (*Compt. rend.*, **107**, 336—339).—From 1 to 10 litres of the water is concentrated by evaporation, and the compounds of iron, magnesium, barium, calcium, strontium, and ammonium, and the silica, carbonic acid, and sulphuric acid are removed by the usual methods. The residue, which consists of alkaline chlorides with probably a small quantity of magnesium chloride, is evaporated to dryness, the liquid being well stirred towards the end of the operation in order to obtain a granular residue. This residue is extracted with alcohol of 90 per

cent., the alcohol distilled off, and the residue evaporated with water acidified with hydrochloric acid, and, whilst still moist, treated with concentrated alcohol, and finally with alcohol and ether. The alcoholic solution is evaporated to dryness, the residue dissolved in 15 to 20 c.c. of water, and treated with ammonium fluoride in the manner already described (preceding Abstract). The lithium fluoride is dried and weighed, and then converted into sulphate and again weighed. This affords a valuable check on the result, since the weight of the sulphate is almost exactly double that of the fluoride, whilst in the case of potassium and sodium the ratio is very different. Should the relation between the two weights indicate that the lithium salt is not pure, it should be redissolved in water and the precipitation repeated.

In order to separate small quantities of magnesium, the sulphate is dissolved in 40 to 50 c.c. of water, and heated with ammonia and sodium phosphate.

In calculating the quantity of lithium dissolved from the fluoride by the washings, 7 c.c. and not 10 c.c. as stated in the previous paper (*loc. cit.*), contain 2 mgrms. of lithium fluoride.

A spring at Maizières, which contained 3·644 grams of solid matter per litre, contained 0·0240 grams of lithium oxide, whilst another at Sautenay contained 8·980 grams of solid matter and 0·0310 of lithium oxide. C. H. B.

Use of Hydroxylamine Hydrochloride in Quantitative Analysis. By A. Lainer (*Monatsh.*, **9**, 533—536).—The author finds that silver nitrate and halogen silver compounds, either in the solid condition or dissolved in sodium thiosulphate or potassium cyanide, are perfectly reduced by treatment with hydroxylamine hydrochloride and alkali. The reduced silver can be readily transferred to a filter and weighed. Results of analyses are given showing the accuracy of the method. G. T. M.

Estimation and Separation of Zinc. By J. Riban (*Compt. rend.*, **107**, 341—343).—The solution is mixed with sodium carbonate until a slight, permanent precipitate is formed, and this is redissolved by addition of a small quantity of hydrochloric acid. An excess of a slightly acid solution of sodium or barium dithionate is added, the liquid diluted so that 100 c.c. contain not more than 0·1 gram of zinc, and it is then saturated with hydrogen sulphide *in the cold*. Under these conditions, zinc sulphide separates as a very dense, granular precipitate, which is readily washed with warm water containing hydrogen sulphide. If the solution is sufficiently dilute, the liberated dithionic acid exerts no appreciable solvent action on the precipitate.

This method is available for the separation of zinc from manganese, iron, and other metals which are not precipitated by hydrogen sulphide in presence of dithionic acid. C. H. B.

Copper Assays. By J. W. Westmoreland (*Chem. News*, **58**, 76—79).—The author does not approve of the fire assay or of the cyanide method; he also disagrees with Peter's strong advocacy of the electrolytic method inasmuch as silver, bismuth, lead, arsenic, tin,

manganese and ferric salts all interfere with it. He advocates E. O. Brown's iodine process, titrating in an acetic acid solution. (Compare Abstr., 1887, 80.) D. A. L.

Detection of Mercury in Urine by Electrolysis. By Ziegeler (*Chem. Centr.*, 1888, 1040, from *Monatsh. pr. Dermatologie*, **7**, 557—558).—150 c.c. of urine is warmed with 1·5 grams of potassium permanganate. When the oxidation is complete, the liquid is filtered and 100 c.c. is treated with 5 grams of sulphuric acid, the liquid shaken to expel carbonic anhydride, and the further examination conducted according to Wolf's method (*Chem. Centr.*, 1888, 867). J. W. L.

The Electrolytic Method as applied to Iron. By E. F. Smith (*Amer. Chem. J.*, **10**, 330—332).—The solution is mixed with an excess of sodium citrate and a little free citric acid; using about 70 to 150 c.c. of liquid and a current liberating 6 c.c. oxhydrogen gas per minute; the deposition generally takes four hours. The separation is complete, the metal is compact, regular, and steel-like, and after washing successively with water, alcohol, and ether, and drying it does not alter when exposed to the air. The presence of aluminium or of titanium does not interfere. A number of test analyses are given. H. B.

Separation of Arsenic, Antimony, and Tin from Gold and Platinum. By L. L. de Koninck and A. Lecremier (*Zeit. anal. Chem.*, **27**, 462—463).—The precipitated sulphides are heated in a stream of hydrogen chloride. Antimony and tin volatilise as chlorides, arsenic as sulphide: gold and platinum remain. M. J. S.

Bromine for Gold Extraction. By W. H. Burfeind (*Chem. News*, **58**, 92).—The use of bromine is suggested as a substitute for chlorine in the laboratory and perhaps in the works also; for the extraction of gold, it has the advantage of requiring no generating apparatus. A weighed quantity of the ore is washed, put in a bottle with bromine-water, agitated from time to time, more bromine added, if required, until there is still an excess of bromine after an hour or so. The precipitate is well washed and the filtrate is treated in the same manner as if chlorine had been used. D. A. L.

Determination of Oxygen in Water. By Blarez (*J. Pharm.* [5], **18**, 55—58).—An estimation can be made in 10 minutes. The following titrated solutions are required:—Sodium hydroxide, one equivalent per litre; ferrous ammonium sulphate, 40 grams per litre, slightly acidified with sulphuric acid; sulphuric acid diluted with an equal volume of water; decinormal solution of potassium permanganate. The apparatus employed is a bromine tube with a bulb capacity of about 250 c.c., the tube of which is cut off 2 to 3 cm. below the stop-cock. Above the bulb is a caoutchouc stopper carrying a cylindrical funnel of about 12 c.c. capacity, having a capillary tube terminating level with the underside of the stopper. The capacity of the tube is measured by filling with water up to the middle of the neck which receives the

stopper and running out the water into a graduated tube; this is only required once for each apparatus. 30 to 35 c.c. of mercury is measured accurately and poured into the tube; 10 c.c. of normal sodium hydroxide is now added and the water to be examined is run in up to the mark in the neck; the volume of water taken is thus known. The stopper with funnel is now thrust into the neck so as to remove all the air; a little water may rise into the funnel, a little mercury run out at the stopcock brings the level down to the bottom of the stopper. Exactly 5 c.c. of the ferrous solution is now placed in the funnel and carefully passed into the tube by running out more mercury until the level of the liquid is again brought to the bottom of the stopper. The liquids are now mixed by giving the tube a gyratory movement, and in five or six minutes the oxygen may be considered to be completely absorbed. There is now added 10 c.c. of sulphuric acid in the same way as the ferrous solution; on agitating the tube, the contents become clear at once. The mercury is now run out, and the aqueous contents are poured out and titrated with permanganate solution. J. T.

Estimation of Glycerol in Crude Glycerin. By R. Benedikt and M. Cantor (*Monatsh.*, **9**, 521—527).—From 1 to 1½ grams of the sample is heated for from 1 to 1½ hours with from 7 to 8 grams of acetic anhydride and about 3 grams of anhydrous sodium acetate, in a wide-mouthed 100 c.c. flask provided with a reflux condenser. After cooling, 50 c.c. of water is added, the flask is again attached to the condenser, and the contents are heated just to the boiling point. By this operation, the glycerol is quantitatively converted into triacetin. The solution is filtered and the amount of triacetin present determined by means of standard caustic soda and hydrochloric acid. Di- and tri-glycerides are decomposed by acetic anhydride in an almost precisely similar way. G. T. M.

Estimation of Glycerol by Oxidation. By V. Planchon (*Compt. rend.*, **107**, 246—247).—When a solution of glycerol is mixed with potassium permanganate and sulphuric acid, there is no appreciable action in the cold, but at 40° carbonic anhydride is evolved, and at a higer temperature oxidation proceeds rapidly and regularly. The carbon is completely converted into carbonic anhydride, and by absorbing this gas in soda-lime, the quantity of glycerol can be accurately estimated. C. H. B.

Determination of Cane-sugar in Liqueurs, Confectionery, and Chocolate. By F. Rathgen (*Zeit. anal. Chem.*, **27**, 433—445).—This may be effected by the use of the saccharimeter. The chief difficulties lie in the preparation of perfectly clear solutions, and in the presence of other optically active substances. Turbid solutions may generally be clarified by shaking with alumina, which has been precipitated from the sulphate by ammonia and thoroughly washed. In the case of chocolate, the further addition of lead acetate and alum is necessary. Coloured liquids should be shaken with blood charcoal. In the absence of inverted sugar or glucose (which may

be known by the absence of reduction when the solution is warmed with copper sulphate and soda), a simple reading (A) in the polariscope, gives by the usual formula the percentage of cane-sugar, but since part of the sugar is generally found to have been inverted by the action of the fruit acids, it is necessary to complete the inversion by heating a portion of the liquid with hydrochloric acid and then to make a second observation, B. If the amount of substance proper to the instrument in use has been taken, the formula $\frac{100(A-B)}{42{\cdot}4-0{\cdot}5t}$, gives the percentage of cane-sugar at the time of analysis, and $\frac{100\ B}{42{\cdot}4-0{\cdot}5t}$, the original percentage. Since the amount of rotation produced by invert sugar is affected by the presence of alcohol, it is necessary in examining liqueurs to evaporate (after neutralising with ammonia) and make up with water. Ammonia should also be added when extracting the sugar from such articles as candied fruits, jellies, and syrups, in all of which much of the sugar will be found to be inverted. The results are given of the examination of numerous liqueurs and articles of confectionery. M. J. S.

Detection of Aldehyde. By W. Windisch (*Zeit. anal. Chem.*, **27**, 514; *Zeit. für Spirit. Ind.*, **9**, 519; *Chem. Zeitung*, **11**, 24).—With metaphenylenediamine hydrochloride, aldehyde forms a reddish-brown, resinous substance, soluble in water and alcohol with blood-red colour and green fluorescence. Highly dilute solutions (1 of aldehyde in 200,000) give a yellow colour, which disappears on treatment with an alkali, and is restored by addition of hydrochloric acid. M. J. S.

Acetone in Urine. By Legal (*J. Pharm.* [5], **18**, 206—207, from *J. Méd. Paris*).—To detect acetone in urine, add some drops of a concentrated solution of sodium nitroprusside, and make the mixture alkaline by the addition of a little potassium hydroxide. A red coloration is produced which disappears after some time; when this has taken place, a little acetic acid is added, and, if acetone is present in the urine, a deep-violet coloration appears. Acetone is found in diabetic urine. Ferric chloride is employed to detect ethyl acetoacetate in urine; in this case, the urine becomes deep-red. The urine of patients who have taken thalline, antipyrine, salicylic acid, and phenol gives the same reaction with ferric chloride, but the colour disappears on boiling in the case of ethyl acetoacetate. If the urine be previously boiled, the colour is not produced in the latter case, but is produced with the other substances. J. T.

Valuation of Crude Sodium Acetate. By G. Neumann (*J. pr. Chem.* [2], **38**, 91—97).—This is a modification of Phillips's method (Abstr., 1886, 747). The dropping funnel has a three-way cock, so that a stream of air may be passed through the apparatus; the air is supplied through a T-piece, so that when it is turned off by the three-way cock it may be used to force the liquid through the funnel, thus preventing the escape by upward pressure from the retort.

The carbonic anhydride is estimated at the same time by connect-

ing the receiver with weighed absorption tubes. The carbonaceous matter in the residue from the distillation is determined by combustion with chromic and sulphuric acids in Classen's apparatus.

A. G. B.

Estimation of Hydrogen Potassium Tartrate and Free Tartaric Acid in Wines. By B. Haas (*Chem. Centr.*, 1888, 1045—1046, from *Zeit. Nahr. Hygiene*, **2**, 97—100).—Two quantities of 50 c.c. each of the wines are evaporated in dishes on the water-bath to about 4 c.c. One of these is, previously to the concentration, half neutralised with potassium carbonate. After the desired concentration is effected, 2 c.c. of glacial acetic acid is added to the half-neutralised portion for each gram of neutralised tartaric acid. 100 c.c. of 95 per cent. alcohol is added to each portion, and the hydrogen potassium tartrate is collected and titrated with alkali. The difference between the amounts of hydrogen tartrate as thus determined corresponds with the free tartaric acid in the wine.

J. W. L.

Test for Uric Acid. By G. Denigès (*J. Pharm.* [5], **18**, 161—162).—The reaction depends on the transformation of uric acid into alloxanic products under the action of nitric acid or of bromine-water, the solution of these compounds in concentrated sulphuric acid, and on the blue colour communicated to commercial benzene by this solution. A little uric acid is placed in a small porcelain capsule with two drops of water and one or two drops of nitric acid, the whole is gently evaporated to dryness, when the mass should be still yellow, or scarcely reddish. It is dissolved in two or three drops of concentrated sulphuric acid, and on adding commercial benzene a beautiful blue coloration appears. The coloration is due to the action of thiophen contained in the benzene obtained from coal-tar on the alloxan and other alloxanic compounds, resulting from the oxidation of uric acid by nitric acid or bromine-water. J. T.

Determination of Fat in Milk, Cream, &c. By C. Schmid (*Zeit. anal. Chem.*, **27**, 464).—In a test-tube of about 50 c.c. capacity, graduated in tenths of a cubic centimetre, 5 c.c. of cream or 10 c.c. of milk is placed; 10 c.c. of concentrated hydrochloric acid is added, and the mixture is boiled with shaking until it becomes dark brown. After cooling, 30 c.c. of ether is added, and the tube is well shaken. The ethereal and aqueous layers separate perfectly clear. The volume of the former is read, and 10 c.c. withdrawn by a pipette is evaporated in a tared basin on the water-bath, whilst blowing upon its surface. The fat is dried at 100° and weighed, and the amount calculated for the whole of the ether solution. The entire operation takes about a quarter of an hour, and gives results of the highest accuracy.

M. J. S.

Analysis of Butter, Oleomargarin, &c. By H. N. Morse and W. M. Burton (*Amer. Chem. J.*, **10**, 322—328).—Hehner's and Koettstorfer's methods of analysis are not capable of distinguishing between good butter and mixtures of butter with certain fats. The following method, which may be made very simple, discriminates in

such cases very easily, and depends on the determination of the total fatty acids, and of those soluble and of those insoluble in water. The following numbers, expressing the percentage of alkali required to neutralise (*a*) the insoluble and (*b*) soluble acids respectively, show the great difference between butter and other fats and oils:—Butter, (*a*) 86·57, (*b*) 13·17; cocoanut-oil, (*a*) 91·95, (*b*) 8·17; ditto washed with water, (*a*) 92·43, (*b*) 7·42; ditto washed with dilute sodium carbonate solution, (*a*) 92·33, (*b*) 7·45; cotton-seed oil, (*a*) 92·05, (*b*) 7·76; oleomargarin, (*a*) 95·40, (*b*) 4·57; lard, (*a*) 95·96, (*b*) 3·82; beef tallow, (*a*) 96·72, (*b*) 3·40. The fat is melted, filtered, and well stirred during solidification; a sample is then saponified with a measured quantity of alcoholic potash, and the excess of the latter determined by hydrochloric acid and phenolphthaleïn; the total acids are thus found. The alcoholic solution is evaporated to dryness and the soap treated with exactly enough hydrochloric acid to liberate all the fatty acids; the liquids are heated in a flask fitted with a reflux condenser until the oil has separated, when it is filtered, and the two sets of acids thus separated are then easily estimated.

H. B.

Action of Sulphur Chloride on Oils, &c. By T. T. P. B. Warren (*Chem. News*, **58**, 4, 15).—Although genuine lard and lard oil yield products with sulphur chloride which are perfectly soluble in carbon bisulphide, yet mixtures of equal parts of cotton-seed oil and lard oil do not, as might be expected, yield a product containing 50 per cent. soluble, in fact, some of the lard oil product appears to be retained by the altered cotton oil. The author now finds that cotton oil does not dissolve when boiled even with concentrated potash; therefore to overcome difficulties like that just noted, the mixed product is saponified with 30 per cent. potash, the glycerol is determined and the fatty acids examined; in this manner, a clue is obtained as to the character of the oils present which yield solid products soluble in carbon bisulphide, but are retained by the insoluble mass. Cases may occur where sulphur chloride yields an insoluble product, but that owing to its solubility in an accompanying soluble fat the insoluble substance may be overlooked; in such cases, a saponification test will also be found useful, as for instance, when cotton oil and lard fat are mixed in such proportions as to yield a soluble sulphur chloride product. The author has succeeded in reproducing cotton oil by heating a mixture of the acids obtained from cotton oil and glycerol in a sealed tube at about 260°, for several weeks; he hence confirms Berthelot's observation on this point; it is noteworthy that the cotton oil acids, unlike the oil, yield with sulphur chloride products soluble in carbon bisulphide. The author points out that the stability of the fatty glycerides in different oils is intimately connected with the drying properties; and the iodine absorption of an oil before and after exposure to the air in a warm place gives an idea of the change effected during oxidation; for example, the iodine absorption of poppy oil exposed in an open dish at 60° fell from 135 to 119 per cent. in 10 days, a sample of rape oil under similar conditions was much bleached, but its iodine absorption only fell 10 per cent. The author attributes

the dark hue of the sulphur chloride products from oxidised oils to the loss of glycerol.

In examining olive oil adulterated with lard and poppy oil, the author determines the total iodine absorption of the mixture, the lard oil is then removed by boiling with moderately strong alkali, supplemented if necessary by washing with ether, then having ascertained the iodine absorption due to the lard oil, it is not difficult to fix on the mixture of olive and poppy oil which yields the residual iodine absorption; the reduction of the iodine absorption of poppy oil by oxidation is an element of uncertainty. The presence of poppy oil may be confirmed by passing ozone through the mixture for a short time; the sulphur chloride product being black and more viscid if it is present. D. A. L.

Detection of Sesame Oil in Olive Oil. By E. Millau (*J. Pharm.* [5], **18**, 169—170, from *Monit-sci.*, 1888).—On treating olive oil directly with sugared hydrochloric acid, a rose colour may be obtained with a perfectly pure oil. Various experiments have shown that the colour in this case is due to the aqueous portion which passes from the fruit pulp when the oil is extracted by pressure. Consequently, to test olive oil for sesame oil, it is necessary to remove this aqueous portion by heating at 110°; then a portion treated with an equal volume of sugared hydrochloric acid in a test-tube gives a rose colour if any sesame oil is present, but remains absolutely colourless if pure. J. T.

Detection of Cotton-seed Oil in Olive Oil. By E. Millau (*J. Pharm.* [5], **18**, 170—171, from *Mont.-sci.*, 1888).—In a porcelain basin of 1000 c.c. capacity, 15 c.c. of the oil in question is heated to about 110°, then, still continuing the heat, a mixture of 15 c.c. of sodium hydroxide of 40° B. and 15 c.c. of alcohol of 92° is slowly poured on to the oil. When the mass becomes homogeneous, 500 c.c. of water is added drop by drop so as to avoid cooling the mass and the formation of lumps. After boiling a short time, the fatty acids are separated by dilute (one-tenth) sulphuric acid. When the acid is in slight excess, about 5 c.c. of the fatty acids is heated gently on the water-bath with 20 c.c. of alcohol to solution, 2 c.c. of silver nitrate solution (30 grams to 100 c.c. water) is added, and the heating continued until about one-third of the liquid has evaporated. The fatty acid remains unchanged if the oil is pure; but if cotton-seed oil is present metallic silver is precipitated. The reaction is very sensitive, indicating 1 per cent. of cotton-seed oil. Sources of error are avoided by thus working on the fatty acids separated by the process. J. T.

Determination of the Oil in Linseed Cake. By R. Klopsch (*Zeit. anal. Chem.*, **27**, 452—457).—It is necessary that the cake intended for extraction with ether should not be dried at too high a temperature or for too long a time. Three hours in the water-oven (at 94—96°) is sufficient to dry the sample thoroughly, and has no influence on the oil determination, but after six hours at that

temperature, and still more markedly at a higher temperature (105°), considerably less oil is extracted by ether. The cause of this is under investigation. M. J. S.

Estimation of Mustard Oil in Seeds of Cruciferæ. By O. Förster (*Landw. Versuchs-Stat.*, 1888, 209—214).—As the various methods heretofore in use for the estimation of mustard oil in seeds are more or less incorrect, the following method has been devised, and used with good results: 25 grams of the powdered substance (oil cake) is made into a thin paste with water, allowed to remain for half an hour, and then heated by the introduction of steam, whereby the oil is volatilised; and may be collected by the usual arrangements of condenser, &c., in a 250 c.c. flask containing 50 c.c. ammoniacal alcohol; when about 150 c.c. of liquid has distilled over, the liquid is allowed to remain for 12 hours in a closed vessel, and afterwards brought to boiling in a beaker, and freshly prepared mercuric oxide is added, sufficient to combine with all the sulphur present. The mixture is then again boiled, and before it is quite cold potassium cyanide is added to remove all excess of mercuric oxide and oxydimercurammonium hydroxide; the weight of the mercuric sulphide obtained is then multiplied by 0·4266, the product representing the mustard oil. The mercuric oxide employed is prepared by decomposing 25 c.c. of a 4 per cent. solution of mercuric chloride with potassium hydroxide and boiling the mixture. A small loss may occur due to the formation of the mustard oil, since potassium myronate in presence of myrosin and water yields besides mustard oil, small quantities of crotonitrile and free sulphur, which remain in the retort; the extent to which this may occur can be estimated by preliminary experiments; sinapolin (diallylcarbamide) carbonic anhydride, and sulphuretted hydrogen may be formed, or diallylthiocarbamide may be produced, but in both cases the hydrogen sulphide simultaneously formed combines with the mustard oil, producing allylamine and carbon bisulphide, or diallylthiocarbamide and carbon bisulphide; the sulphur is not lost, as with alcoholic ammonia it forms ammonium thiocarbonate, which yields mercuric sulphide with mercuric oxide. E. W. P.

Tests for Antipyrine and Antifebrine, and Fahlberg's "Saccharin." By D. Lindo (*Chem. News*, **58**, 51).—The author suggests the following reaction as a test for "saccharin." Not less than 0·5 mgrm. solid "saccharin" is evaporated to dryness over a water-bath with excess of nitric acid, and whilst still on the bath, a piece of potash and a few drops of water, or better still, of 50 per cent. alcohol are added; colour develops immediately, and by inclining the dish streaks of blue, violet, purple, and red flow from the potash.

Antipyrine is heated with concentrated nitric acid over a flame until reaction commences, the flame being then removed. When the action ceases, a fine purple liquid remains, which on adding water and filtering yields a purple-red solution and a violet precipitate.

A minute quantity of antifebrine is strongly heated with a little pure concentrated sulphuric acid, the product when cool is mixed with

water and with some highly dilute nitrite solution, and may then be tested like ordinary sulphanilic acid by means of γ-naphthol, thymol, or phenol. D. A. L.

Estimation of Emetine. By A. Kremel (*Arch. Pharm.* [3], **26**, 419, from *Pharm. Post*, **21**, 151).—10 grams of ipecacuanha root, finely powdered, is rubbed up with the same amount of potassium hydroxide and water, dried on a water-bath, finely powdered, and extracted with hot chloroform. After evaporation of the nearly colourless solution, the emetine, which is slightly yellow, is dried at 100° and weighed. The average amount obtained is 1·80 per cent. Flückiger's method, extraction with ammoniacal chloroform, does not yield the emetine sufficiently pure to give the value of the ipecacuanha root. J. T.

Filtering "Crude Fibre" and Silver Chloride. By T. B. Osborne (*Chem. News*, **58**, 90—91).—The following plan of filtration is suggested as rapid and efficient for crude fibre determinations. Fold the paper to form a cone of somewhat smaller angle than that of the funnel, place in position on the platinum cone, moisten and apply suction; the paper generally tears down a fold, forming a narrow rift from vertex upwards with a loose network of fibres across it. The liquid and fibre to be filtered are stirred up and thrown on this filter; filtration proceeds rapidly and well.

When there is any difficulty in filtering silver chloride from solutions containing organic matter, the greater part of the liquid should be passed through a Gooch filter, the precipitate should then be dissolved in slight excess of ammonia, reprecipitated with nitric acid, and washed by decantation; it can then be thrown on the filter without fear of passing through. D. A. L.

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748	20	from bottom		*for*	"C. Fischer"	*read*	"O. Fischer."

VOL. XLVIII.

744	17	„	top	„	"isosaccharic acid"	*read*	"isosaccharinic acid."
„	9	„	bottom				
„	26	„	top	„	"isosaccharate"	„	"isosaccharinate."
745	9	„	„				
„	18	„	„	„	"metasaccharate"	„	"metasaccharinate."
„	14	„	„	„	"trioxyadipic"	„	"trihydroxyadipic."
1013	10	„	bottom	„	"creasote"	„	"creosote."
1342	col. ii, 21	„	„				
1078	18	„	top	„	"Hantsch"	„	"Hantzsch."

VOL. L.

631	9	„	bottom	„	"*Ber.*, 9"	„	"*Ber.*, 19."
1012	17	„	top	„	"585"	„	"885."
1013	col. ii, 17	„	bottom	„	"1040"	„	"1014."
1033	14	„	„	„	"ortholotylaniline"	„	"orthotolylaniline."

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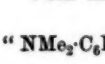
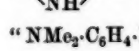
139	6	„	„	„	"$NMe_2 \cdot C_6H_4$  $C_6H_2Me \cdot NH_2$"	*read*	"$NMe_2 \cdot C_6H_4 \cdot N$  ."
437	20	„	„	„	"*Ann. Chim. Phys.*"	*read*	"*Ann. Phys. Chem.*"
651	12	„	„	„	"$C_{15}H_{16}NO_4$"	„	"$C_{13}H_{16}N_4O$."
727	3	„	top	„	"Hentzsel"	„	"Hantzsch."
737	2	„	bottom	„	"Valem"	„	"Valeur."
738	21	„	top	„	"carbamide"	„	"carbamine."
976	6	„	bottom	„	"methylketone"	„	"methylketole."
1070	20	„	„	„	"Günwald"	„	"Grünwald."

ERRATA.

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Page	Line						
34	19	from	bottom	*for*	"H. SAUER"	*read*	"A. SAUER."
39	13	„	top	„	"β-phenylacrosazones"	*read*	"-phenylacrosazones."
46	12	„	bottom	„	"mannite"	„	"mannitol."
67	3	„	„	„	"Abstr., 1882, 92"	„	"Abstr., 1883, 92."
123	20	„	„	„	"S. A. PŘIBYTEK"	„	"S. PŘIBYTEK."
154	3	„	top	„	"Diphthalic acid"	„	"Diphthalylic acid."
188	top line			„	"N. REALI"	„	"N. REALE."
248	7	„	„	„	"fatty nitroketones"	*read*	"the hydrogen sodium sulphite compounds of fatty nitrosoketones."
267	13	„	bottom	„	"sugar"	„	"sugars."
294	5	„	top	„	"T. BOUCHARDAT"	„	"G. BOUCHARDAT."
357	9	„	„	„	"A. MAASEN"	„	"A. MAASSEN."
379	23	„	„	„	"Quinine alkaloïds"	„	"Cinchona alkaloïds."
388	24	„	„	„	"nitric acid"	„	"nitric oxide."
415	13	„	„	„	"H. W. WARREN"	„	"H. N. WARREN."
424	23	„	„	„	"Trans., 1886, 379"	„	"Trans., 1886, 708."
426	1 & 4	„	„	„	"Platoso-"	„	"Platino-."
429	21	„	bottom	„	"H. TAUFS"	„	"H. TAUSS."
456	19	„	top	„	"A. MOSSO"	„	"U. MOSSO."
466	17	„	„	„	"Isebutylphenylenediamine"	*read*	"Isobutylphenylenediamine."
483	12 & 13	„	bottom	„	"skatole" *read* "scatole."		
492	8	„	„	„	"$OH{\cdot}C_6H_2Me{<}\begin{smallmatrix}CO\\CH\end{smallmatrix}{>}C_6H_3{\cdot}OH$" *read* "$OH{\cdot}C_6H_2Me{<}\begin{smallmatrix}CO\\CH_2\end{smallmatrix}{>}C_6H_3{\cdot}OH$."		
493	13	„	„	„	"triacetyl"	*read*	"tetracetyl."
557	20	„	„	„	"R. WAGNER"	„	"R. F. WAGNER."
617	14	„	top	„	"J. ROSENHEIM"	„	"T. ROSENHEIM."
646	16	„	bottom	„	"CLANDON"	„	"CLAUDON."
679	10	„	top	„	"Mononaphthene"	„	"Nononaphthene."
693	2	„	bottom	„	"S. RACINE"	„	"(?) S. RAEINE."
745	23	„	top	„	"secretion"	„	"aëration."
761	22	„	„	„	"29—30°"	„	"20—30°."
775	11	„	„	„	"NADESCHIDIN"	„	"NADESCHDIN."
827	22	„	„	„	"hexabenzoic"	„	"hexazobenzoic."
834	15	„	„	„	"A. SCHEUK"	„	"A. SCHENCK."
894	12	„	bottom	„	"T. GERLACH"	„	"G. T. GERLACH."
916	26	„	top	„	"R. G. GRISSON"	„	"R. G. GRISSOM."
924	16	„	„	„	"C. HERSH"	„	"C. HERSCH."
971	14	„	„	„	"1883"	„	"1885."
1005	9	„	„	„	"37"	„	"31."
1103	7	„	bottom	„	"JACCKLE"	„	"JAECKLE."
1035	16	„	top	„	"MeO"	„	"MgO."
1051	22 & 26	„	„	„	"hexamethylene"	„	"hexamethylenamine."
1067	21	„	bottom	„	"Ethylmalonate"	„	"Ethyl Malonate."
1128	14	„	„	„	"Berques"	„	"Bergues."
1141	4	„	„	„	"coils"	„	"cells."
1203	26	„	top	„	"Dihydrazanthraquinone"	*read*	"Dihydroxyanthraquinone."

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